# Feasibility of Using Ohmsett for Dispersant Testing and Research


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# Acknowledgment

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# 1. Introduction

## 1.1 Study Rationale and Statement of Problem

One difficulty in using oil spill dispersants in the field is predicting and measuring the effectiveness of the dispersant over a range of conditions. The main variables include oil type and weathered state, dispersant type, dispersant-to-oil ratio, application method, slick thickness, and sea state. Dispersant testing in small-scale laboratory tests has been shown to be of limited value in predicting dispersant effectiveness in actual spill situations. Dispersant testing in ocean field trials has yielded useful results, but these are only valid for the narrow range of conditions that were experienced in the trials. Testing a wide range of variables at sea in many trials is possible, but would be expensive and also difficult to get approved by regulators. The situation remains that little quantitative information is available on dispersant effectiveness under real spill conditions. What is needed is a facility that can be used to test a wide range of dispersant-use variables under conditions that simulate actual conditions much better than existing laboratory tests do. Ohmsett may be such a facility.

The problem is that Ohmsett is not currently equipped to do dispersant testing. There are at least four concerns:

1.  that dispersant entering the tank water from one or more tests may affect the results of subsequent dispersant tests;

2.  that dispersant in the water may affect slick behavior in subsequent tests involving spill recovery or containment devices;

3.  that turbidity caused by dispersed oil from one or more tests may affect underwater viewing of subsequent tests; and

4.   that removal of dispersant and tiny dispersed oil droplets from the water might be difficult and expensive to accomplish.

Before Ohmsett can be used for routine dispersant testing and research, it is necessary to assess these technical concerns.

## 1.2 Study Objective and Goals

**Objective.** The objective of the study was to examine various ways that dispersant testing at Ohmsett might be accomplished and to evaluate their feasibilities and costs. At the outset it was realized that the main job would be to investigate and find a cost-efficient and fast method of filtering the oil droplets and dispersant out of the tank water.

**Goals.** The study approach involved seven goals or tasks to reach the objective of the study. These are outlined below.

**Task 1 – Review of Past Dispersant Testing and Current Limitations at Ohmsett.** The purpose of this task was to review the handful of dispersant tests that were performed at Ohmsett in the past, and to review the current government regulations related to the discharge of contaminated water from the tank.

**Task 2 – Interfacial Tension Tests.** The purpose of this task was to determine the critical concentration of dispersed oil and dispersant that could affect subsequent tests involving dispersant or subsequent tests involving boom and skimmer work.

**Task 3 – Turbidity Tests.** The purpose here was to determine the critical concentration of dispersed oil that would cause enough turbidity in the tank to prevent underwater viewing and photography of the dispersant tests.

**Task 4 – Initial Design of Dispersant Tests.** The main purpose of this task was to determine how many dispersant experiments could be done before the critical concentrations identified in Tasks 2 and 3 are realized. This would indicate how often the tank will have to be treated to remove dispersant products and dispersed oil. To accomplish this task properly, it was necessary to scope out a design for a dispersant program at Ohmsett.

**Task 5 – Tests to Assess the Effectiveness of Current Filter System.** The purpose of this task was to determine whether the current filtering material used at Ohmsett, namely, diatomaceous earth, is able to remove mechanically dispersed oil, chemically dispersed oil, and dispersant chemicals themselves.

**Task 6 – Tests to Identify Other Filter Materials and Approaches.** In the event that the current filter system was found to be unable to filter dispersant and/or dispersed oil, the purpose of this task was to research and test other filtering materials and approaches.

**Task 7 – Full Scale Testing at Ohmsett.** Finally, the results of the above tasks are put to the test and verified at full-scale at the Ohmsett facility itself.

The following chapters present details of each of the tasks in the order presented above.

# 3. Current Operations and Past Dispersant Testing at Ohmsett

It is useful to review current operations at Ohmsett, focusing on aspects that pertain to the feasibility of doing dispersant testing there. Subjects of interest include the mechanical features of the facility, such as: methods available for discharging oil and dispersant; in-water sampling and viewing capabilities; the salinity of the basin water; methods and standards for maintaining water clarity; and government restrictions on discharging the tank contents into New Jersey waters. It is also useful to review the history and assess the relevance of any dispersant testing that has taken place at Ohmsett since the facility began operations in 1974.

## 3.1 Description of Ohmsett

The Minerals Management Service operates the National Oil Spill Response Test Facility known as Ohmsett (Oil and Hazardous Materials Simulated Environmental Test Tank) located on the grounds of the U.S. Naval Weapons Station Earle, in Leonardo, New Jersey. Ohmsett is used primarily for the testing and development of devices and techniques for the control and cleanup of oil spills.

The main structure of the facility is a pile-supported concrete tank with the following water dimensions: 203 meters (667 feet) long, 20 meters (66 feet) wide, and 2.4 meters (8 feet) deep (see Figure 1). The tank is filled with brackish water from nearby Sandy Hook Bay. At the time of this study the salinity was maintained at a level of approximately 17 parts per thousand (ppt). (The Ohmsett staff now maintain the tank water at a salinity of 30 - 35 ppt to more closely resemble ocean conditions.)

The tank is spanned by three movable bridges. The main bridge is equipped with an oil distribution system capable of laying oil slicks on the surface several meters ahead of the devices being tested. Devices can be towed at speeds up to 6.5 knots.
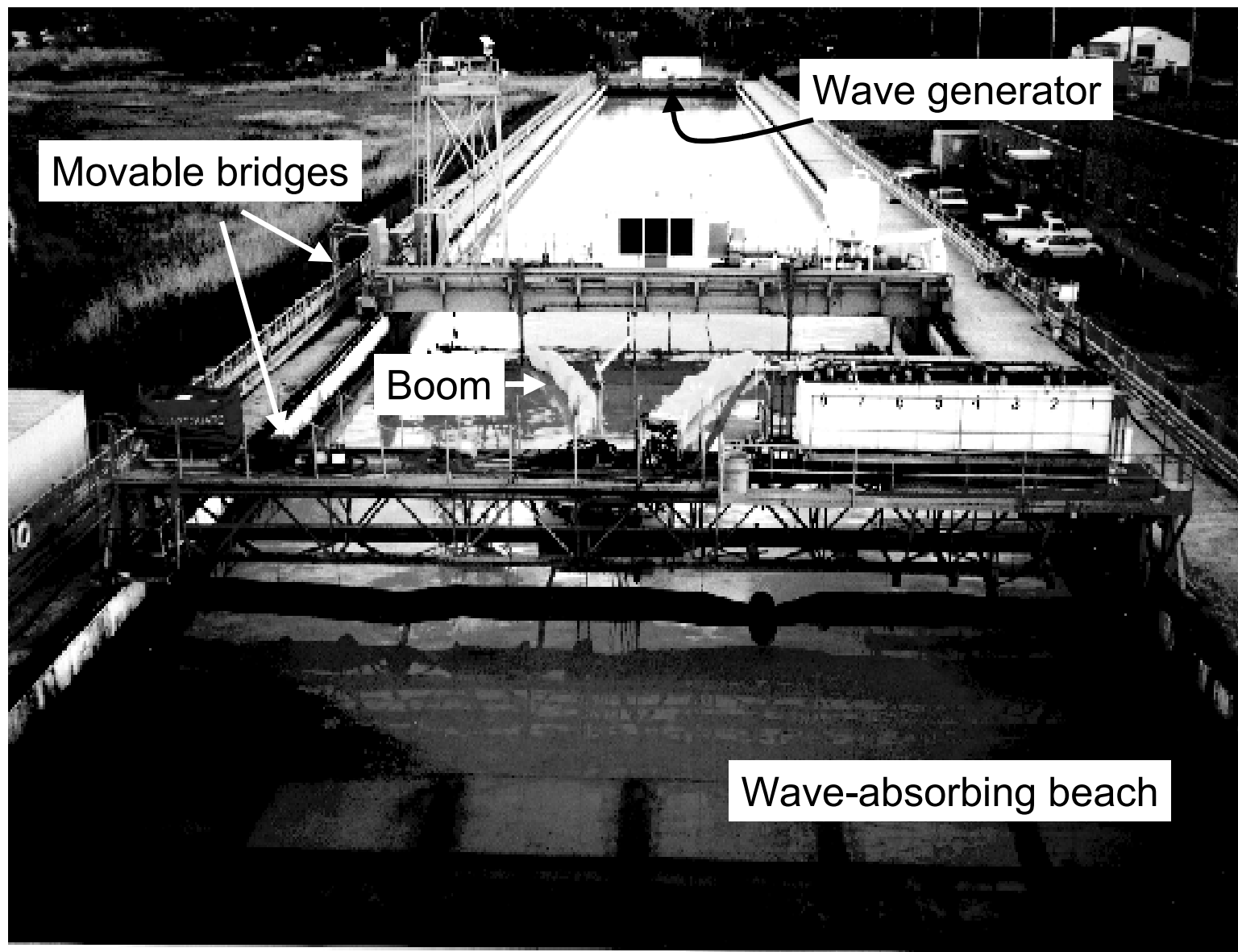
**Figure 1. The Ohmsett Wave Basin as Viewed from the North End (Boom Under Test)**

The tank includes a flap-type wave generator at the south end and a wave-absorbing beach at the north end which can be lowered to the bottom of the tank to allow waves to reflect from the north wall.

The wave generator can produce regular (unidirectional sinusoidal) waves up to 61 centimeters (2 feet) high and up to 45 meters (100 feet) long. With the beach lowered a "confused condition" can be produced which resembles a "harbor chop" with heights of 70 centimeters (2.3 feet).

The clarity of the water in the Ohmsett tank is achieved by the use of a leaf-type filtration system consisting of nylon leaves pre-coated with diatomaceous earth (DE). Suspended solids in the size range of between one to one hundred microns (micron = one-millionth of a meter) can be removed by the filter. (The filter system is described in further detail in Section 7.1.) Algae growth is controlled through the use of an electrolytic chlorinator.

Other features of the tank include:

- a control tower with a computerized 32-channel data collection system as well as above- and below-water video;

- a centrifuge system to recycle recovered test oil;

- blending tanks with a water and oil distribution system to produce custom oil/water emulsions for testing;

- permanent and mobile storage tanks that can hold over 227,000 liters of test fluids;

- a vacuum bridge to clean the bottom of the tank; and

- a shop area for special fabrication.

## 3.2 Discharge Regulations

The operators of Ohmsett are allowed to discharge the tank contents into nearby Sandy Hook Bay according to the conditions outlined in Permit No. NJ00057762 issued by the New Jersey Pollutant Discharge Elimination System (NJPDES) Regulations N.J. A.C. 7:14A-1 et seq.

On the understanding that the filtered water from the tank could be discharged up to twice per year, the permit from the New Jersey Department of Environmental Protection states, among other conditions not relevant to this study, that:

1. there shall be no discharge of floating solids or visible foam in other than trace amounts;

2. there shall be no visible sheen;

3. the daily maximum of total suspended solids shall not exceed 20 mg/L;

4. The daily maximum of petroleum hydrocarbons shall not exceed 15 mg/L (approximately 15 ppm by volume); and

5. The daily maximum pH shall not exceed 9.0.

There is no restriction on salinity, nor is there any apparent restriction on concentrations of detergent or other surfactants in the water, except, as noted above, that no visible foam must be evident in the discharge.

## 3.3 Past Dispersant Testing at Ohmsett

Ohmsett started in business in 1974 and since then about 200 official projects have been accomplished. Of these, 12 involved dispersants in some way. For interest, these are listed in Table 1.

**Table 1. Dispersant-Related Projects at Ohmsett from 1974 to Present**

| Job Order Number | Title | Dispersants used in Tank? |
|---|---|---|
| 24 | EPA, Coastal Waters, 5-bar Gates | yes |
| 38 | EPA, Harbor Oil Spill Dispersant Spraying System | no |
| 57 | EPA, Warren Springs Laboratory 5-bar Gates | no |
| 70 | Elf/Aquitaine, Skimmer/Dispersant System | no |
| 102 | EPA, Dispersant– In Tank Model | no |
| 111 | EPA, Lab Dispersant Tests | no |
| 117 | EPA, Improved Dispersant Effectiveness Tests | no |
| 118 | EPA, Dispersant Equipment– Field Tests | no |
| 120 | EPA, Calibration and Installation of Dispersant Application Equipment | no |
| 134 | EPA, Field Dispersant Effectiveness Tests | no |
| 137 | EPA&API, Calibration and Evaluation of a Dispersant Application System | no |
| 146 | Evaluation of Improved Laboratory Dispersant Effectiveness Test | no |

It is noted that of the 12 projects listed only one actually involved using dispersant in the tank. Most were either tests of dispersant application equipment without the use of dispersant, or laboratory studies performed in the lab space of the facility. The report of the one experiment involving the use of dispersant substances, however interesting, does not provide any information on the subjects of interest in this study, namely, how the tank was cleaned subsequent to the tests, whether the tests could be viewed underwater or whether there was any concern about how the tests might affect subsequent tests involving booms and skimmers.

To gain more information, two persons who worked at the tank in the late 1970s and early 1980s were interviewed. These were Mr. Rich Griffiths and Mr. Mike Borst. According to them, at least

two unofficial scoping studies were accomplished at the facility in which dispersants were used in the tank. Three new facts were learned from the interviews and these are reported as follows.

1. For much of the history of Ohmsett operations (at least during the long period of employment of Mr. Borst and Mr. Griffiths) the filter system was operated with diatomaceous earth as the filter aid. Occasionally, cellulose was combined with the diatomaceous earth when surface active materials in the tank had to be filtered out.

2. In one test involving the application of dispersant onto an oil slick, it was noticed (through the underwater viewing ports) that the oil was not dispersing homogeneously throughout the tank, but rather the slick tended to sink to a certain depth and remain there. This experiment was carried out after a lengthy period of non-use of the tank, and it was surmised that the tank was thermally stratified due to its standing without mixing. This is a lesson for the future. If dispersant work is planned, the tank should be well mixed prior to the beginning of work.

3. In one experiment, to remove dispersant from the tank, it was decided to stop the chlorination process for a period to allow natural bacterial action to decompose the dispersant. This was proved to be successful but was very messy. After the tank chlorination was stopped the algae proceeded to grow quickly and voluminously, and to sink to the bottom of the tank. This created substantial extra work for the filter operators and for those responsible for having to vacuum the bottom and clean the rest of the tank. After the filtering and the cleaning, however, the dispersant was found to be removed, as measured by surface tension analysis.

# 4. Effects of Dispersant in Ohmsett Water on Surface Slick Behavior

The concern of both previous and current Ohmsett operators with performing dispersant testing at the facility is that dispersant left in the tank's water after a given test might affect the properties and behavior of floating oil in subsequent tests, either more dispersant tests using crude oils or conventional tests using the three Ohmsett test oils. The specific concern is that the interfacial tension of floating oil might be significantly lowered and thus produce premature slick entrainment (in conventional boom tests) or enhanced slick dispersibility (in dispersant effectiveness tests). To understand this issue, two sets of experiments were performed. First, laboratory tests were conducted to measure the surface tension and interfacial tensions of the water and oils involved, first using regular Ohmsett water and then using the same water dosed with dispersant. Second, a standard dispersant effectiveness test procedure was used in the laboratory to check the effects of dispersant-dosed water on the dispersibility of surface slicks.

## 4.1 Interfacial Tension Tests

Tests were performed to measure: (1) the surface tension of Ohmsett water and the surface tension of the test oils and crude oils; and (2) the interfacial tension between Ohmsett water and the test and crude oils, both as a function of dispersant concentration in Ohmsett water. The following describes the test materials, methods and results of the laboratory study.

### 4.1.1 Test Materials

**Oils.** The three test oils used at Ohmsett to test skimmers and booms were selected for obvious reasons. These are called Calsol, Sundex, and Hydrocal. Also, two crude oils that are transported in large volumes in U.S. waters were selected. These are Alaska North slope crude oil and Arabian Light crude oil. The properties of the five oils are presented in Table 2.

**Table 2. Properties of Oils used in Study**

| Oil Type | Interfacial Tension, dynes/cm | Surface Tension, dynes/cm | Specific Gravity @15° C | Viscosity, cP | |
|---|---|---|---|---|---|
| | | | | 10°C | 25°C |
| Alaska NS | 17.6 | 32.7 | 0.880 | 24 | - |
| Arab Lt | 28.6 | 32.5 | 0.870 | 20 | - |
| Calsol | 26.6 | 35.8 | 0.933 | 11,400 | 1700 |
| Sundex | 30.0 | 37.0 | 0.946 | 160,000 | 18,000 |
| Hydrocal | 21.0 | 34.0 | 0.905 | 1000 | 150 |

**Dispersants.** Two dispersants were selected for study: Corexit 9527 and Corexit 9500. These are widely available in the U.S.

**Water.** Ohmsett water itself was used in all tests. This was shipped from the facility to the SL Ross Laboratory in a 55-gallon drum, which turned out to be somewhat rusty on the inside, so the water used in the tests required a coarse filtering to remove rust particles. The salinity of the water was measured to be only 10 ppt. This is substantially less than the value of 17 ppt that is indicated in Ohmsett literature.

### 4.1.2 Test Apparatus and Procedure

All surface tension and interfacial measurements were made using the CSC DuNouy Ring Tensiometer, model 70545 in general accordance with the procedure outlined in ASTM D971-82.

### 4.1.3 Results of Interfacial Tension Tests

Figures 2 through 8 present graphs depicting the results of the study, which are discussed in turn. The unit of mN/m in the graphs is identical to dynes/cm.

**Surface Tension of Ohmsett Water Figure 2).** The addition of dispersant (both Corexit 9527 and 9500) in concentrations as low as 1 to 2 ppm (vol) had a noticeable effect on the surface tension of Ohmsett basin water. A concentration of 50 ppm dispersant approximately halved the surface tension of the water. During the testing it was noted that concentrations of dispersant as low as 10 ppm caused the water to foam when it was vigorously agitated, and a concentration of 50 ppm produced stable foam on the water surface that survived for a considerable time after the agitation ceased.

**Surface Tension of Floating Oils (Figures 3 through 8, top curves).** The addition of dispersant to Ohmsett basin water had no appreciable effect on the surface tension of any of the five oils tested. Surface tension of floating oil is measured by testing at the oil/air interface. The results suggest that the dispersant surfactants, otherwise located in the water and at the oil/water interface, are not present in appreciable concentrations in the body of the oil or at the oil/air interface.

**Interfacial Tension of Hydrocal (Figure 3).** The presence of very small concentrations of dispersant in Ohmsett water (in the range of 2 to 5 ppm) was enough to produce a measurable drop in interfacial tension of the Hydrocal test oil. A interfacial tension reduction of approximately 50% was found at concentrations of 50 ppm. Corexit 9527 appeared to reduce the interfacial tension slightly more than Corexit 9500. Reductions in interfacial tension to below 18 mN/m falls outside the range currently required for standard Ohmsett boom tow tests.

**Effect of Interface Aging (Figure 4).** In tests with Hydrocal, the interfacial tension decreased further over time, but not sharply, after the oil was placed on Ohmsett water containing concentrations of 10 ppm of either 9527 or 9500.

**Interfacial Tension of Calsol (Figure 5).** The addition of dispersant to Ohmsett water reduced the interfacial tension of Calsol, but not by as much as with Hydrocal.

**Interfacial Tension of Sundex (Figure 6).** The addition of 5 and 10 ppm dispersant to Ohmsett water produced a marked reduction in the interfacial tension of Sundex. As with the other oils, a reduction in interfacial tension of approximately 50% was measured at 50 ppm.

**Interfacial Tension of Alaska North Slope Crude Oil (Figure 7).** The presence of dispersant in Ohmsett water reduced the interfacial tension of fresh Alaska North Slope crude oil, but not by as much as with the Ohmsett test oils. The effect was measurable at 10 to 20 ppm and caused an approximately 40% reduction at 50 ppm.

**Interfacial Tension of Arab Light Crude Oil (Figure 8).** The addition of dispersant to Ohmsett water reduced the interfacial tension of fresh Arab Light crude oil, more so than with Alaska North Slope. The effect was measurable at 2 to 5 ppm and caused an approximately 50% reduction at 50 ppm. As with the Hydrocal oil, the 9527 dispersant had a slightly greater effect than the 9500.

In addition to the above, it was noted in the experiments that the presence of dispersant changed the spreading behavior of the two crude oils; neither produced a sheen on the water with dispersant concentrations as low as 1 ppm. As well, preliminary qualitative tests showed that, although the presence of Corexit 9527 in Ohmsett water did not appear to affect the oil sorption of a commercial sorbent pad at concentrations of 50 and 100 ppm, it did reduce the hydrophobic character of the sorbent considerably. The sorbent dipped in Ohmsett water containing 50 or 100 ppm of dispersant retained considerably more water than the one dipped in un-dosed Ohmsett water. Similar visual tests with an aluminum plate indicated that the addition of dispersant to the water at 50 and 100 ppm slightly reduced the amount of oil adhering to the plate.

### 4.1.4 Summary and Major Implications of Results

The results clearly indicate that the presence of dispersant in Ohmsett water strongly affects the interfacial tension of the three Ohmsett test oils and the two crude oils tested. Even concentrations as low as a few ppm are enough to cause dramatic effects. This does not necessarily mean that such small concentrations will affect the behavior of slicks during boom and skimmer performance tests, but there is a reasonable possibility of this for the following reason. During boom testing at Ohmsett one of the standard measurements is to note the minimum tow speed at which the slick becomes unstable and small droplets are broken off the main slick and swept under the boom. This first indication of oil entrainment is called "first loss". It is possible that very small dispersant

concentrations in the water, in reducing the interfacial tension between the oil and water, will cause slicks to become unstable and entrained before they normally would. Whether this will indeed happen is simple enough to confirm by tests at Ohmsett itself, but it is difficult to confirm in the laboratory because of scaling problems. Further, even if full-scale tests were to show a negligible effect with low dispersant concentrations, a negative perception could exist with clients of Ohmsett, which would be unacceptable. Therefore it must be assumed that even small concentrations of dispersant in the water are unacceptable for conventional equipment testing at Ohmsett, and that the tank will have be thoroughly cleaned and cleared of dispersant following a dispersant testing program.

The question remains whether the results of a dispersant effectiveness test in the tank will be affected by low concentrations of dispersant in the tank caused by a previous dispersant test. If this were the case, the tank would have to cleaned and cleared of dispersant after each experimental run, and this could prove to be expensive and unfeasible. Fortunately, this hypothesis can be tested in the laboratory by the use of a standard dispersant effectiveness test, as discussed next.

## 4.2 Dispersant Effectiveness Tests

In all standard dispersant effectiveness tests, the procedure involves creating a slick of oil on a contained amount of water, adding dispersant to the oil and then mixing the system in some way to create an oil-in-water dispersion. For the current requirement this procedure is changed by adding dispersant to the water phase and not to the oil phase. In this way one can assess the extent to which the dispersant in the water from one test at Ohmsett might enhance oil-in-water dispersion in the next.

### 4.2.1 Test Materials

Fresh Alaska North Slope crude oil was used in all tests. The properties of the oil are shown in Table 2. Ohmsett water was used in the tests, after filtering small particles of rust. The salinity was about 10 ppt. The dispersant used was Corexit 9500.

**4.2.2 Test Procedure**

Tests were performed using the WSL Revolving Flask Test (also called the Labofina Test). This popular test is described in a number of sources. Appendix A includes a copy of the test description from Nordvik et al. 1993. Briefly, the standard test works as follows. Dispersant in the amount of 0.2 mL is added dropwise to 5 mL of test oil on the surface of 250 mL of sea water in a conical separatory funnel. The funnel is rotated for 2 minutes, stopped and allowed to rest for 1 minute. Then 50 mL of the contents are drained, removed and analyzed using a spectrophotometer for oil concentration to yield a dispersant effectiveness value, measured as the amount of oil in the water phase divided by the starting amount oil, expressed as a percentage.

As indicated above, the test was modified in one major way: the dispersant used in the test was added to the water phase and not to the oil phase. The oil was poured on the surface after dispersant was mixed with the water. Two series of tests were performed. The first series again involved a small change to the test in that 100 mL was withdrawn for oil extraction and spectrophotometric analysis and not 50 mL as outlined in the test procedure. This was done to make the test more sensitive to low concentrations of oil in the water phase, in anticipation of the dispersant effectiveness results being low. In the second series the standard 50 mL were withdrawn. Tests were conducted at room temperature, 19.5°C for the first series and 25°C for the second.

**4.2.3 Results of Dispersant Effectiveness Tests**

The results, shown in Table 3, indicate that there is virtually no difference between the control (natural dispersion with no dispersant) and the dispersant-in-oil cases up to at least 400 ppm. A noticeable effect occurs at 800 ppm where the dispersant effectiveness was measured to be 27.6 percent.

Note that when the same of amount of dispersant used in the 800 ppm experiment was used in a standard test (but applied onto the floating oil), the dispersant effectiveness was much higher, namely 41.6 percent. This is not surprising because one of the dispersant surfactants (namely, sodium

**Table 3. Dispersant Effectiveness Using Modified WSL Test (Dispersant Initially in the Water)**

| Dispersant Concentration | Dispersant Effectiveness, % | |
| --- | --- | --- |
| | First Series (100 mL withdrawn)[a] | Second Series (50 mL withdrawn)[b] |
| Control, no dispersant | 6.0 | $2.7 \pm 1.9$ |
| 20 ppm in water | 2.1 | - |
| 80 ppm in water | 4.9 | $5.0 \pm 1.1$ |
| 250 ppm in water | - | $6.0 \pm 1.0$ |
| 400 ppm in water | - | $6.1 \pm 0.7$ |
| 800 ppm in water | 27.6 | - |
| Dispersant-on-Oil Test (equivalent to 800 ppm) | 41.6 | - |

a. No replications

b. Three replications; results expressed as average ± one standard deviation

diiso-octyl sulphosuccinate (Aerosol OT, Alcopol O)) is known to be particularly effective only when added directly to the oil. Finally, it is interesting to note that the dispersant effectiveness values in Table 3 are somewhat lower that the values that would normally be expected with the WSL test using an oil like Alaska North Slope crude and Corexit 9500. The reason is that Corexit 9500 is designed for normal sea salinity situations (in the 30 ppt range). The product is relatively ineffective in fresh water and only moderately effective in brackish waters (the Ohmsett water used in the test had a salinity of only 10 ppt).

**4.2.4 Summary and Implications of Results**

The results show that dispersant concentrations in the water can reach at least 400 ppm before having a noticeable effect on the results of a standard dispersant effectiveness test. In the 2.6 million gallon tank at Ohmsett, 400 ppm of dispersant represents 1040 gallons of dispersant. If, for example, dispersant experiments at Ohmsett were to involve a drum of oil (55 gallons) each, and a dispersant-

to-oil ratio of 1-to-25 is used, the amount of dispersant used in each experiment would be about 2 gallons. This means that hundreds of such experiments could be done consecutively, with increasing dispersant concentrations in the tank, without concern that the dispersant in the tank were having an effect on the dispersant effectiveness results.

The probable reason that no effects were found on dispersant effectiveness at the surface – even with in-water concentrations in the 100 ppm range – is that these concentrations are still orders of magnitude lower than dispersant concentrations at and near the water surface during a dispersant application. When dispersant is applied to an oil slick in a dispersant-to-oil ratio of 1-to-25, the concentration of dispersant in the floating oil is 40,000 ppm. This high concentration would tend to overwhelm any effect that the dispersant in the water might be having.

In summary, there should be no concern of performing a series of dispersant tests with a modest concentration of dispersant in the water. As will be shown in Chapter 6, the main problem is not the high dispersant concentration but the high oil concentration.

# 5. Turbidity/Visibility Study

## 5.1 Introduction

The obvious objective of dispersant effectiveness testing is to obtain quantitative information on the subject of dispersant performance. Nevertheless it is useful for experimental design purposes, and perhaps educational or training purposes, to be able to view the experiments from an underwater vantage point. In this regard, one advantage of dispersant testing at Ohmsett compared to testing at sea is the ability to use the viewing panels on the tank's side to observe the underwater plume of dispersed oil as it develops over time.

The objective of this study was to determine the likely visibility in the Ohmsett test tank after a chemical dispersant effectiveness test. Depending on the amount of oil used in each test and the amount dispersed, oil concentrations generated in the tank could be as low as 10 ppm (for 26 gallons dispersed) and as high as 100 ppm for experiments involving 4 or 5 drums of oil. The question is: If the oil were to remain suspended in the tank, how would this affect one's ability to see the formation of a dispersed oil cloud in subsequent tests?

Two sets of experiments were designed to answer the question. The goal of the first set of tests was to generate a calibration curve to relate the concentration of oil in water to the water turbidity, using a HF Scientific DRT 100B turbidimeter. This is the same device employed routinely at Ohmsett to monitor the tank's water quality; when the turbidity reaches an unacceptable level the filtration system is put into service to clarify the water. The information generated in this set of tests provides a link between water clarity measured during routine Ohmsett tests and that measured after chemical dispersion tests.

The second test was designed to provide an estimate of the distance that an observer could see into the test tank at different dispersed oil concentrations. The test methods and results for the two tests follow.

## 5.2 Oil Concentration vs. Turbidity Results

Oil concentration versus turbidity curves were developed for two oils, Alaska North Slope crude oil and Arab Light crude oil. The oils were pre-mixed with Corexit 9527 dispersant (at a 1:50 ratio) and 0.25 mL were mixed with 250 mL of filtered Ohmsett water to create an initial oil concentration of 1000 ppm. The water was filtered four times prior to use to remove the rust coloring that had developed in the water during shipping from the Ohmsett facility to the SL Ross Laboratory. The 1000 ppm sample was then diluted to 500, 150, 100, 50 and 10 ppm and the turbidity of each was measured using the HF Scientific DRT 100B turbidimeter. The results were recorded in nephelometric turbidity units (NTUs) and are shown in Table 4 and Figure 9.

**Table 4. Turbidity Measurements for Dispersed Oil Samples**

| Oil Concentration (ppm) | Turbidity (NTUs) for Specified Oil Types | |
|:---:|:---:|:---:|
| | Arab Light Crude | Alaska North Slope Crude |
| 1000 | 750 | 865 |
| 500 | 388 | 480 |
| 250 | 144 | 152 |
| 100 | 78 | 80 |
| 50 | 23 | 48 |
| 10 | 14 | 28 |
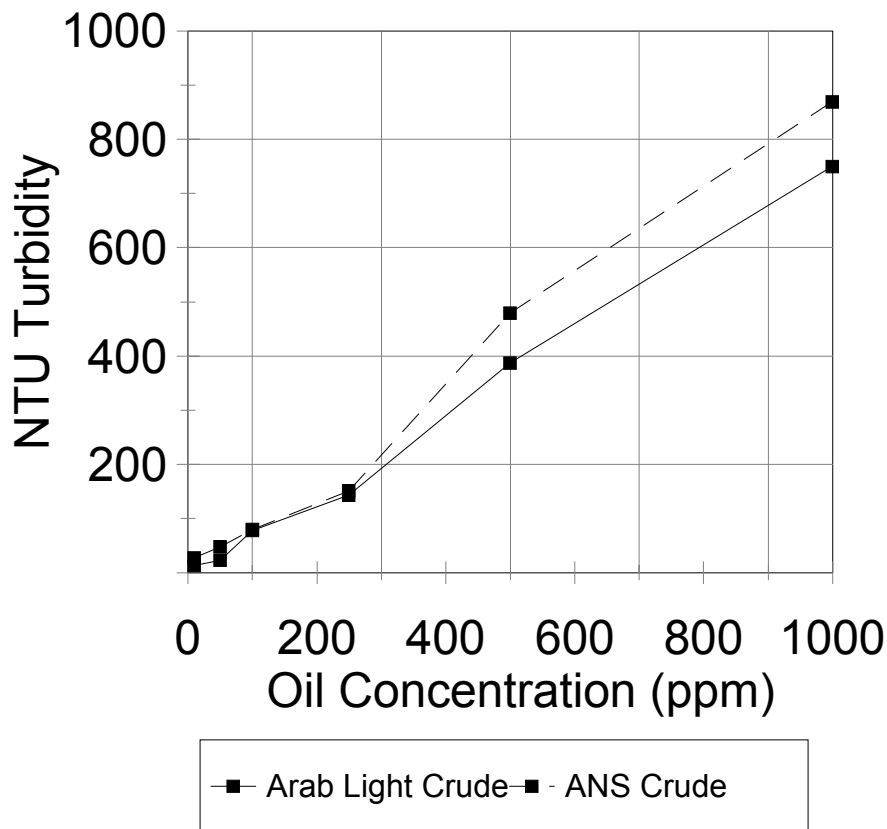
**Figure 9. Turbidity versus Oil Concentration**

## 5.3 Visibility vs. Turbidity

Two 6-foot lengths of 4-inch diameter, clear acrylic tubing were fitted end to end to make a 12 foot long cylinder. One end was capped and fitted with a drain hose. The tube was mounted vertically against a building and filled with water from the roof. Figure 10 shows this setup. A 2-inch diameter steel washer was colored black, in alternate quarters, to provide a visual target that was lowered into the tube using a clear fishing line. In successive tests, the tube was filled with fresh tap water, 32 ppt salt water, salt water with oil at 5 ppm concentration, and salt water with oil at 20 ppm concentration. The oil used was Arab Light crude and it was dispersed into the water using Corexit 9527 at a dosage of 1:50 and vigorous mixing. The distance that the visual target could be lowered

into the tube from above and still be seen was measured for each of the different tube contents. The results are shown in Table 5.

It is evident from this test that even very small oil concentrations generated from a dispersant effectiveness test will reduce the visibility in the tank such that the behavior of oil on subsequent tests will not be visible from the viewing ports. Oil concentrations of 5 ppm clouded the water in the tube to the extent that objects 2 feet into the liquid could not be seen. Visibility in the Ohmsett tank could be even worse than that recorded in this simple test since light will have to penetrate through a larger contaminated water depth to illuminate the viewing area. It is possible to verify this supposition by reviewing past records of turbidity readings taken at Ohmsett. This is done in the next section.
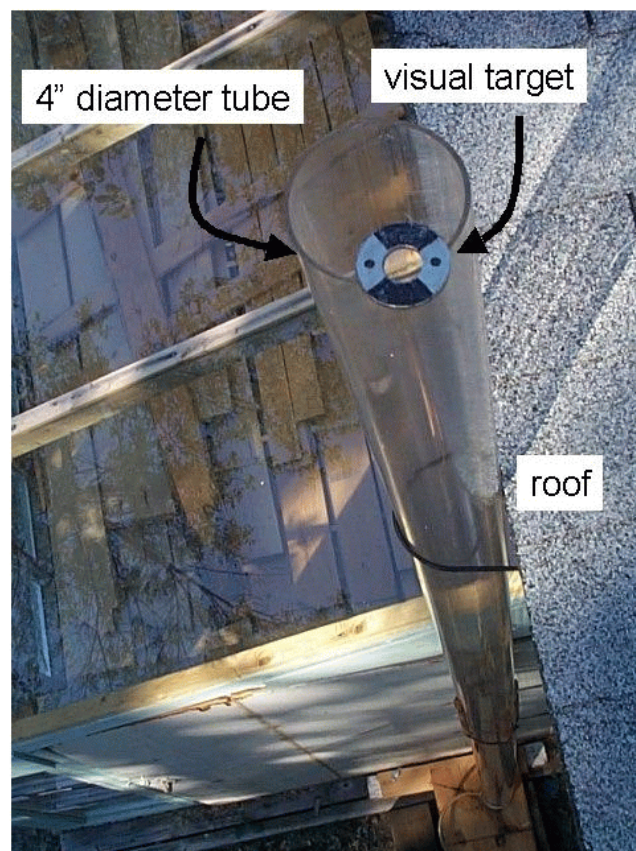


**Figure 10. View from Rooftop of 12-ft Vertical Tube for Measuring Visibility**

**Table 5. Visibility Limits**

| Tube Contents | Visibility of Disk (distance in inches) | Turbidity (NTU) |
|---|---|---|
| Fresh tap water | 120 (full tube length) | 0.25 |
| 32 ppt salt water[a] | 52 | 7.5 |
| 5 ppm oil in salt water | 23 | 12 |
| 20 ppm oil in salt water | 11 | 22 |

a. Salt used was a rather impure, agricultural grade

## 5.4 Turbidity Data from Ohmsett

Information on Ohmsett turbidity is available in the operating instructions for the Ohmsett filtration system (MAR 1997). Figure 11 graphically shows the turbidity of influent and effluent water samples that were taken from the Ohmsett filter during a particular, unidentified season of tests (MAR 1997).

The graph indicates that the turbidity of the tank water (the influent) on average was in the range of 0.2 to 2 NTUs, although several periods involved readings much higher than this. The graph does not suggest any correlation between NTU value and visibility, but some information on this is available from two underwater photo/video studies that were conducted in the 1970s, namely the "Ohmsett Underwater Video Study" in 1975 and the "NAVFAC Water Clarity Tests" in 1976. Unfortunately, these reports have been lost, but in MAR 1997 a reference to the reports indicates that poor visibility of as little as four feet was realized with a turbidity of 3.4 NTU. Excellent visibility was shown in excess of 65 feet at mid tank when influent turbidity was 0.2 NTU and effluent turbidity was 0.02 NTU. These low values, however, were virtually never attained in the operating season represented by the data in Figure 11.

These numbers correspond well to the numbers attained in the present experimental study.
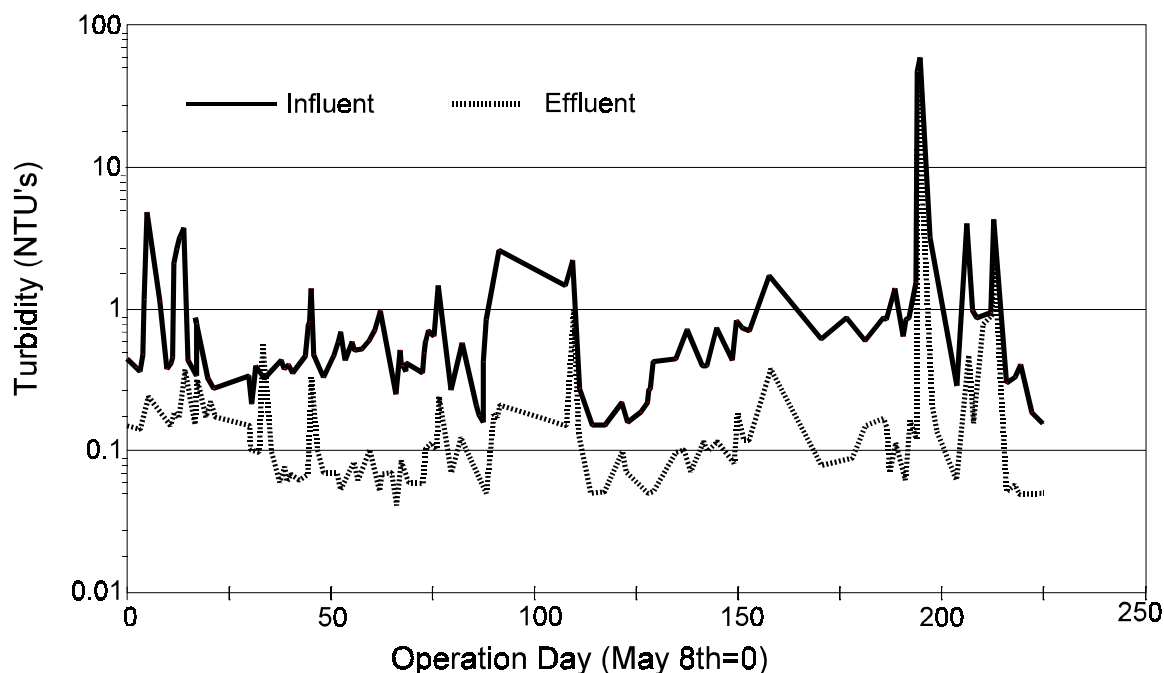
**Figure 11. Turbidity of Ohmsett Filter Influent and Effluent during One Full Season**

## 5.5 Summary and Implications of Turbidity/Visibility Work

The results of the above experimental study and past Ohmsett studies indicate that underwater oil behavior effects at Ohmsett will only be visible by keeping turbidity to very low levels. If dispersed oil concentration in the tank reaches a value of only a few ppm, it will be impossible to view underwater effects. As discussed in the next chapter each dispersant experiment will certainly lead to these concentrations and higher. Therefore, in a series of dispersant experiments, only the first experiment will be visible underwater and be recordable by video or photography. For example, if three experimental runs are done per day and the tank is thoroughly cleaned in preparation for the next runs, then one in three experiments will be visible underwater. This is not ideal, but may be acceptable considering that the qualitative benefits of underwater viewing are much less important than the quantitative aspects of the experiments.

# 6. Scoping Out a Dispersant Testing Program at Ohmsett

The specific purpose of this task is to determine how many dispersant experiments at Ohmsett can be done consecutively before critical dispersant and oil concentrations in the tank are realized. This will indicate how often the tank will have to be treated to remove dispersant products and dispersed oil. To accomplish this task properly, it is necessary to scope out a design for a dispersant program at Ohmsett. This will serve as a framework within which key problems can be analyzed.

## 6.1 Background and Statement of Problem

To review, the results of the interfacial tension tests showed that very low concentrations of dispersant in Ohmsett water (as low as 5 ppm) are enough to cause a measurable drop in interfacial tension between floating oil and the water. It is assumed that this would be unacceptable with respect to the testing of booms and skimmers at Ohmsett because there is a risk that a significant drop in interfacial tension will promote early entrainment of a slick under the containment boom and will also negatively affect oleophilic skimming efficiency.

For the Ohmsett tank which contains $2.6 \times 10^6$ gallons of water, 5 ppm translates into 13 gallons of dispersant, or 325 gallons of oil [for a DOR of 1:25] (See Table 6).

This means that after dispersant testing, the tank would have to be filtered of dispersant before proceeding to a test program involving booms or skimmers, if the dispersant test program involved 325 gallons of oil or more, and this amount of oil were present as dispersed oil in the tank.

This need to clear the tank of dispersants does not apply to dispersant testing, however. The results of the WSL dispersant testing program indicates that dispersant effectiveness testing of oils in the Ohmsett facility will likely not be affected until the concentration of dispersant in the water reaches 400 ppm or higher. In this case, several dispersant tests could be done in sequence without concern

**Table 6. Calculations of Oil and Dispersant Concentrations in Ohmsett Tank**

| Oil Amounts, gallons | Dispersant Amounts[a], gallons | Oil Concentration in tank, ppm | Dispersant Concentration in tank, ppm |
|---|---|---|---|
| 20 | 0.8 | 7.7 | 0.3 |
| 40 | 1.6 | 15.4 | 0.6 |
| 60 | 2.4 | 23.1 | 0.9 |
| 80 | 3.2 | 30.8 | 1.2 |
| 100 | 4.0 | 38.5 | 1.5 |
| 120 | 4.8 | 46.2 | 1.8 |
| 140 | 5.6 | 53.8 | 2.2 |
| 160 | 6.4 | 61.5 | 2.5 |
| 180 | 7.2 | 69.2 | 2.8 |
| 200 | 8.0 | 76.9 | 3.1 |
| 220 | 8.8 | 84.6 | 3.4 |
| 240 | 9.6 | 92.3 | 3.7 |
| 260 | 10.4 | 100.0 | 4.0 |
| 280 | 11.2 | 107.7 | 4.3 |
| 300 | 12.0 | 115.4 | 4.6 |
| 328 | 13.1 | 126.1 | 5 |
| 26,000 | 1,040 | 10,000 | 400 |

a. Dispersant-to-oil ratio = 1-to-25

that the dispersant in the tank water was having an influence on the effectiveness of dispersant applied to a test slick of oil. The amount of oil that could be spilled to yield 400 ppm of dispersant (at a DOR of 1-to-25) is 26,000 gallons. (This translates into an oil concentration in the water of 10,000 ppm.)

This large number seemingly suggests that – aside from the problem of underwater viewing, which will only be possible in the first experiment of a series – many dispersant experiments could be done in sequence at Ohmsett without having to filter oil and dispersant from the water. However, the analysis does not take into account the fact that dispersant effectiveness at Ohmsett will likely be determined primarily by measuring oil concentrations in the water immediately beneath treated, dispersing slicks. The concentrations of interest could be as low as 100 ppm; therefore, background

levels of oil in the tank must be significantly lower than the anticipated concentrations of dispersed oil. Clearly, allowing the background concentrations of oil to reach levels approaching this number are not acceptable.

There are two main conclusions that arise from this:

1. It is likely that a series of dispersant tests can proceed at Ohmsett, but without the capability of underwater viewing of all tests. A number of tests could be done in sequence, without concern that the dispersant in the tank were affecting dispersant effectiveness results, but the concentration of oil in the tank could not be allowed to build up to a level that interferes with the quantitative measurement of dispersed oil beneath the treated slicks; and

2. The tank would have to be thoroughly cleaned of dispersant (and oil) before starting a test series involving booms or skimmers.

In summary, the main problem in running a series of dispersant experiments is not the amount of dispersant that will be put into the tank, but the amount of oil. A high concentration of dispersed oil in the tank, say in the 100 ppm range, is likely to be too high a background concentration for measuring dispersed oil concentration in the next dispersant effectiveness test. To determine acceptable background levels, and to understand other important factors, it is useful to scope out how dispersant testing at Ohmsett could be done, as is done next.

## 6.2 Review of Variables That Could Be Investigated At Ohmsett

Several large and expensive offshore oil spill experiments, conducted in the North Sea in the 1990s, attempted to measure dispersant effectiveness (a summary of the trials is in SL Ross 1997). Most of the important variables (such as sea state and oil type/weathered state) were investigated in one or more of the trials, although the variables were not altered in a systematic way, and the experiments had to be carried out within the logistic, safety and technical constraints imposed by working at sea.

There are two clear advantages of doing dispersant testing at Ohmsett rather than at sea: (1) the ability to alter and control variables in a systematic way; and (2) the much lower costs of the testing. These are the same advantages that have justified the activity at Ohmsett in the area of boom and skimmer testing.

As with boom or skimmer testing, the main idea with dispersants is to do testing with a view toward predicting product performance in actual spill conditions over a range of conditions. The main variables to consider for dispersant testing include:

1. Oil type and weathered state
2. Dispersant type
3. Dispersant-to-oil ratio
4. Application method
5. Slick thickness
6. Sea state

The environmental variables of air/sea temperature and salinity are not easily altered at Ohmsett, but could be considered under special circumstances.

Each of the variables listed above is considered in turn.

### 6.2.1 Oil Type and Weathered State

At least two different oil types should be part of the test program. These would be carefully selected to show the potential differences in dispersant response. There is little point in using the three Ohmsett test oils (Sundex, Calsol and Hydrosol) as these are synthetic blends formulated for use in boom and skimmer testing. Of more relevance to dispersant testing would be one or more crude oils and/or a series of fuel oil blends that meet desired viscosity targets.

Should crude oils be used they will need to be weathered prior to use to better reflect real-world conditions and provide a more stable test fluid. The current thinking is that weathering the crude oils in the Ohmsett tank itself will not be a good idea, at least in the beginning. A two-step process is recommended: (1) to use artificially weathered oils until the protocols and difficulties in the testing procedure are ironed out; and then later (2) to consider weathering oils in the tank using procedures currently being proposed under another research project at Ohmsett.

Having to pre-weather crude oils before they are put into the Ohmsett tank will certainly detract from the 'realism' of the tests, but this may be a small sacrifice considering the other benefits gained, as mentioned above. Weathering the oils artificially and creating emulsion by using mixing other than wave action will yield an emulsion product that bears an uncertain resemblance to that formed at sea, but it may have to be done, at least initially.

A second test oil to consider would be a series of IFO-380 residual fuel oils 'cut-back' to varying viscosities with heavy gas oil (MDO - Marine Diesel Oil). Such fuel oil dispersibility testing is popular in Europe at this time. Producing a series of fuel oils with a wide range of viscosity, and hence a wide range of WSL dispersant effectiveness results (with a particular dispersant and dose rates), and then testing these at Ohmsett would have practical usefulness, topicality and relevance on its side. Further, it would lead to a standard set of test fluids that will be relatively easy to obtain and unvarying over time, which would allow comparative testing of various dispersant products.

**6.2.2 Dispersant Type**

At this time there seems little point in testing anything other than Corexit 9527 and Corexit 9500 at the Ohmsett facility, except under contract to a client interested in such.

**6.2.3 Dispersant- to-Oil Ratio**

Until the experimental protocols are ironed out, the usual dispersant-to-oil ratio (DOR) of 1:25 should be used. It must be recognized, however, that this DOR is based on very little information

and there are a number of experts who disagree with it. Combined with studies on different oil slick thicknesses (see below) this could be a very worthwhile variable to be investigated at Ohmsett. DORs of 1:50 and 1:100 would be worth looking at. Again, the results of the work would need to be benchmarked against findings from lab studies, past and future.

**6.2.4 Application Method**

The main advantage of Ohmsett over laboratory systems in terms of dispersant application is that spray systems can be tested at full scale. This does not mean that aircraft need to be used to apply dispersant, but that the spray nozzles and pumps used in the full-scale systems can be used at the Ohmsett trials. This is a major advantage because the drop size distribution of the dispersant spray is likely an important parameter.

Virtually all experts recommend that dispersant should be sprayed neat without dilution onto slicks, and this is the recommendation for the initial studies at Ohmsett. However, there is a renewed interest in fire monitors and the use of water-diluted dispersant (that is, the dispersant is educted into the main water flow, as is fire fighting foam). The comparison of neat application versus water-diluted application would be an interesting subject for Ohmsett research in the future. One way of assessing the effectiveness of different kinds of application would be to compare the result with oil pre-mixed with dispersant.

**6.2.5 Slick Thickness (and Spill Volumes to Consider at Ohmsett)**

Slick thickness is a crucial variable. Thicknesses measured at recent field trials in Europe suggest that even small spills will have an emulsion thickness (of the thick portion of the slick) in the order of 1 to 5 mm, much of it due to windrowing. To create a slick that is 1 mm thick over the entire area of the tank (4000 m$^2$), one would need 4 m$^3$ or 1060 gallons. This is much oil, and when all of this is put into the tank, it would create a dispersed oil concentration of about 410 ppm. This concentration would definitely be too high for a background level, and would have to be significantly reduced before proceeding to the next test.

The idea, rather, would be to create a slick of reasonable and realistic thickness (say, 1 mm thick oil or proportionately more for emulsified oil) but of area (and volume) that is the least that one can use and still obtain realistic results. Current thinking is that the following dimensions are credible and workable. Using the moving bridge, a slick could be laid out that is 1 mm thick, about 2 meters wide and about 50 meters long. This represents a volume of $0.1 \text{ m}^3$ or 26 gallons. If dispersant is applied to this slick and the slick is completely and instantaneously dispersed into the top 1 meter of water, it would produce a dispersed oil concentration of 1000 ppm. Gradually, after some time the oil would mix in the whole tank and produce a maximum concentration of 10 ppm.

If dispersant effectiveness were only 25%, this would produce an initial upper-meter concentration of 250 ppm, and a final homogeneous concentration of 2.5 ppm.

The above scenarios suggest that several dispersant tests could be done sequentially. In these the upper meter or so would be sampled in each test yielding concentrations perhaps in the 250 to 1000 ppm range, with background concentrations growing steadily from zero ppm oil to 7.5 to 30 ppm just before the fourth experiment. This signal-to-background ratio of 33.3 (1000/30 or 250/7.5) seems acceptable, but it would be lower in practice because the dispersion process would not be instantaneous (yielding lower initial concentrations in the upper layer of water) and because the dispersed oil would take some time to mix homogeneously to the 7.5 to 30 ppm levels noted above.

Pre-weathering the oil, with evaporation and emulsification, should render it of high enough viscosity to remain as a coherent carpet if it were laid down from the moving bridge. The oil/emulsion viscosity would have to be in the order of several thousand cP and the bridge should be moving at about 2 knots (1 m/s) for this to happen. The oil/emulsion could be pumped out of a 2 meter long spreader bar (a tube with holes drilled every couple of inches) at a suitable rate to produced the required thickness over the 50-meter length of the experimental slick. In this way 0.5 mm, 1 mm and 2 mm slicks should be possible.

The dispersant should be applied after the discharged oil has had a chance to stabilize on the surface, but before the oil has had a chance to spread out. This could be achieved by mounting a dispersant

spray bar (also 2 meters wide) on the same moving bridge. It could be positioned 'aft' of the moving bridge if the oil deposition bar is mounted ahead, as sketched below. If it were seen in this set-up that the slick was not stabilized before applying dispersant, the other moveable bridge could be brought into play to apply dispersant after a short delay, or the dispersant pass could wait until the bridge returned quickly to the starting position after the oil deposition run.
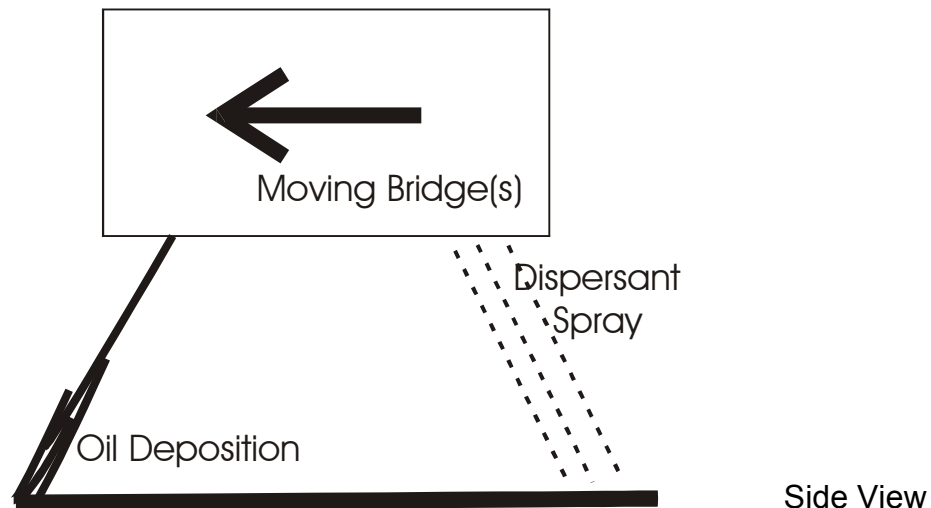


## 6.2.6 Sea State

The relationship between dispersant effectiveness and sea state is very important to understand, and yet, because of a lack of quantitative field data, it is also poorly understood. Because Ohmsett does well in simulating a range of sea state conditions, testing at the facility offers a large advantage over laboratory testing and even testing in the field where the weather and sea states are always an uncertainty.

### 6.2.7 Temperature and Salinity

Water and air temperatures at Ohmsett are obviously not easily controllable, but testing in the fall and early winter is possible, and the results of such testing can be compared to the results collected during the warmer months.

At the time of the study Ohmsett operators maintained a salinity of approximately 17 ppt which is that of the bay water. If dispersant testing at Ohmsett is found to be feasible and there is a desire to proceed, it will be necessary to increase the salinity of the tank to sea water conditions. This is because the main interest is using dispersants on marine spills at sea where full salinity conditions exist, and the dispersant products of particular interest today are of the salt water type, namely Corexit 9527 and 9500.

One idea, then, is to consider testing the effect of salinity on dispersant effectiveness during this initial period when the salinity of the tank will have to be increased. The advantage is that it is easier to add salt to water than to remove it. The disadvantage is that this area of research (assessing the effect of salinity on dispersant effectiveness) is not the highest priority, and perhaps the first series of tests should involve the assessment of more important variables, such as dispersant dosage, sea state and oil type and viscosity, and a benchmarking of the results against sea trials.

## 6.3 Suggested Dispersant Test Program At Ohmsett

### 6.3.1 Experimental Variables

From the foregoing description the possible variables are:

**Test oils:**
1. Weathered heavy crude oil (to be determined)
2. Weathered medium crude oil (to be determined)
3. 'Cut-back' IFO-380 residual fuel oil to resemble

1. IFO-60   (intermediate fuel oil having a kinematic viscosity [cst] at 50°C)
2. IFO-120
3. IFO-180
4. IFO-380

**Test dispersants:**

1. Corexit 9527
2. Corexit 9500

**Treatment rates:**

1. DOR of 1:25
2. DOR of 1:50
3. DOR of 1:100

**Slick thickness:**

1. 2 mm
2. 1 mm
3. 0.5 mm

**Simulated sea state:** To be decided

**6.3.2 Measurements to Make**

**Visual/Video**. Visual observations, recorded on video, will probably be made useless by the turbidity of the dispersed oil in the water, except for the first and possible second experiment in a series. They might make interesting viewing and might be of educational or training value, but will not be of much quantitative use.

**Flow-through UV Fluorescence**. The dispersed oil in water concentration should be measured with Turner AU10 fluorometers with the 'short' oil filter set. The fluorometers use an excitation frequency of 254 nm and an emission frequency of 360 nm. They are regularly used (when fitted with other filter sets) to monitor water quality and should be available for short-term rental as needed. They will require data loggers. If six fluorometers are available, it is recommended that the submersible pumps, which are used to convey continuously sampled water to the fluorometers, be

placed along the tank centerline at 1 and 2 meters depth and two to either side (also at two depths) to monitor the sideways dispersion of the plume, as shown below:

Surface



Side View
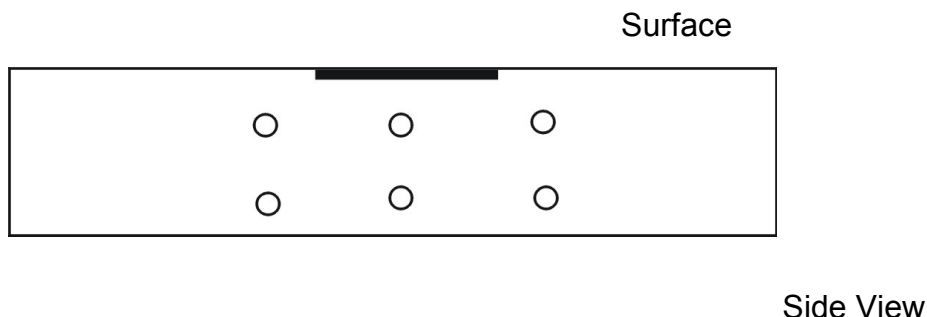
With suitable filters and calibration these fluorometers can measure from well below 0.5 ppm up to 100 ppm dispersed oil. The readings will need to be calibrated with water samples that are taken and extracted with solvent.

An accurate mass balance will always be difficult. Initially there will be a very low level of dispersed oil due to natural dispersion. When the dispersant is added this will rapidly rise. Eventually, if allowed to, the dispersed oil will be evenly dispersed throughout the entire volume of the water in the tank; 53 gallons will give 20 ppm overall. However, before total mixing is achieved there will be much higher readings in the plume under the slick. It will not always be possible to measure this accurately because the scale of the 'patchiness' of oil concentration with distance would require an large number of fluorometers. Accordingly, one of the most important things to be measured would be the 'average' rate of increase in oil concentration in the water, with the transient excursions smoothed out.

One might try to use sorbent to collect and measure all of oil remaining on the surface after an experiment (to check or calculate dispersant effectiveness values), but this is known to be difficult to do. Besides, the result would just be one point on a graph, i.e., a measurement at the conclusion of the test. UV Fluorescence (UVF), which provides measurements throughout the test, will provide a more detailed picture.

### 6.3.3 Experimental Approach

The experimental design will require serious analysis and preliminary testing, but the following are some ideas on how testing might proceed.

Under quiescent conditions and with the use of the moveable bridges, oil is discharged in the center of the tank to form a rectangular shape, 2 m x 50 m. The UVF underwater system takes a background reading of oil concentration in the "test zone" under the slick. The wave generator is started. Dispersion begins and is recorded on a continuous basis by the UVF system. After some time the experiment is stopped. This could be when the remaining slick on the surface reaches the sides of the tank (to avoid wall effects) or it could be when the rate of dispersion has leveled off to some low number, or could be based on some other criteria. In any case, say that this time is in the order of ½ hour to 1 hour.

After the experiment the wave generator is stopped and the dispersed oil is allowed to rise to the surface for a period of time (see Section 6.5 below). Say this is 1 or 2 hours. The oil on the surface would be carefully sorbed or skimmed, leaving some dispersed oil in the tank that is relatively low in concentration and inhomogeneously distributed. The wave generator is turned on for a certain amount of time (say ½ hr to 1 hr) to mix the contents of the tank as thoroughly as possible. Perhaps an air bubbling system could be installed on the bottom of the tank to enhance mixing. The wave generator is turned off and the tank is ready to proceed to the second experiment, this time with a higher background reading in the test zone. The entire experimental cycle takes 2 to 4 hours, depending mostly on the time allowed for the dispersed oil to rise to the surface after the waves are stopped. This would then allow two to four experiments per 8-hr working day.

During the remaining 16 hours of the day, the filter would continue to work removing as much oil and dispersant as possible. At the start of the next working day any surface oil would be skimmed, and then the wave generator (and air bubbler) would be started to mix the tank again as thoroughly as possible before the beginning of the next series of experiments.

This would continue every day until either the background concentration of dispersed oil becomes too high or the series of experiment is over. At this point the tank water would be thoroughly cleaned and filtered in preparation for other test programs.

## 6.4 Removing Dissolved Dispersant Chemicals

Corexit 9500 and 9527 both contain about 40% to 50% surfactants. About 5% is sulphosuccinate (Aerosol OT, Alcopol O) and the rest is a blend of Span and Tween nonionic surfactants. If one can take out the dispersed oil produced in each test with a filter-like mechanism, the majority of the nonionic surfactant should go with it as well, theoretically. The small amount of sulphosuccinate left will certainly not affect subsequent dispersant tests.

Even if quite a few tests were done in a series – say 20 – with 100 liters (26 gallons) of oil per test, this would use a total of 2,000 liters of oil and 80 liters of dispersant (at a DOR of 1:25). Of the 80 liters of dispersant, about 40 liters would be nonionic surfactants, and much of this should be removed with the oil.

If UVF is used as the primary analytical tool, one could only do a few tests before cleaning the water, as discussed earlier. A background of 40 ppm when trying to measure an overall increase of 20 ppm, albeit with peaks up to 100 ppm, is about as much as is tolerable. Therefore, with 10 to 20 ppm increases to the water body after each experiment (depending on spill volumes used), one is limited to two to four experiments before having to implement a water clean-up operation.

If most of the oil and the majority of the nonionic surfactants were removed in the clean-up, only 250 to 500 mL of sulphosuccinate would be left in the nearly 10,000 tonnes of water each time. If the sulphosuccinate could not be removed, it would build up in the system. After 20 tests the tank would have only 5 to10 liters of sulphosuccinate in nearly 10,000 tonnes of water, i.e., 0.5 to 1 ppm sulphosuccinate. This can be considered to be an insignificant amount that will have no effect on any testing program at Ohmsett.

## 6.5 Removing Oil from the Tank Prior to Filtering

It is suggested above that one experiment involving 26 gallons of oil (0.1 m³) when fully dispersed and distributed in the tank will create a concentration of 10 ppm. The next experiment will bring the concentration up to 20 ppm, and so forth, until finally the level becomes too high, requiring a filtering program to lower the concentration. It must be recognized that this is the worst case because (1) not all oil will be dispersed in each experiment, so the remainder will be recoverable by skimming systems, and (2) much of the oil that remains dispersed during simulated oceanic mixing will rise to the surface after the wave generator is stopped and the basin becomes quiescent, and therefore will also be recoverable.

When dispersant is applied onto slicks in the field, a wide drop size distribution of dispersed oil is formed. Some drops are relatively large and rise quickly to the surface and others are small enough to stay dispersed in the water under average sea state conditions. Studies (Lunel 1993, Lunel 1995) have indicated that droplets having diameters in the size range of 50 to 100 μm (μm = $10^{-6}$ m) tend to represent most of the volume of dispersed oil sampled deep under treated slicks. The question is: If oceanic mixing (or the wave generator at Ohmsett) is stopped, how long will it take drops of this size to rise to the surface?

The rise of rigid oil droplets in water is governed by Stokes Law, in which the terminal rise velocity is related to the main variables by the equation:

$$U = g \cdot d^2 \cdot (\rho_o - \rho_w) / 18 \, \mu \quad \text{(For Re > 0.3, which is always the case in this example)}$$

where

$U$ = terminal velocity, m/s

$g$ = acceleration due to gravity, = 9.8 m/s²

$d$ = droplet diameter, m

$\rho_o$ = oil density = say, 850 kg/m³

$\rho_w$ = water density = 1008 kg/m³

$\mu$ = viscosity of water = 0.001 Pas

Plugging in the numbers yields

$$U = 8.6 \times 10^4 \cdot d^2$$

Using this equation, we can calculate rise velocities of drops of certain diameter and thus determine the time needed for the drops to rise through a given height. The calculations are shown in Table 7.

**Table 7. Time Needed for Dispersed Drops to Rise Through 1 Meter and 2 Meters of Water**

| Drop diameter, μm | Rise Velocity, cm/min | Time to Rise 1 m, hrs | Time to Rise 2 m, hrs |
|---|---|---|---|
| 10 | 0.052 | 32 | 64 |
| 20 | 0.20 | 8.2 | 16 |
| 50 | 1.2 | 1.3 | 2.6 |
| 100 | 5.2 | 0.32 | 0.64 |

It is seen that most of the dispersed oil volume in the Ohmsett wave basin will rise and surface within about 2 hours, assuming that the volume-average drop size is in the 50 to 100 μm range. This oil can be easily skimmed. Remaining in the tank will be very fine particles smaller than 20 μm or so. Although these represent little oil volume, they probably will inhibit underwater visibility even in very low concentration.

In summary, it seems likely that much dispersed oil can be removed physically following a dispersant experiment. The advantage of this is that it allows more experiments to be run in a given period of time and reduces oil loadings on the filter. At some point, however, there will still be a need to filter the water to remove all oil and dispersant.

## 6.5 Summary of Scoping Analysis

This scoping analysis suggests that a number of dispersant experiments could be run in sequence at Ohmsett before having to filter the body of water to remove dispersed oil. Perhaps two to four experiments could be run in a row involving about 26 to 53 gallons of oil each.

The main issue remains how, and how quickly, one can remove the dispersed oil from the system. This will affect the number of tests that can be accomplished per week, the overall costs of the testing program, and the days needed after the program to start a boom or skimmer program. This crucial issue – the capability of the filter in removing dispersant and dispersed oil – is the subject of the next chapter.

# 7. Diatomaceous Earth and Cellulose as Filters for Oil and Dispersant

The main purpose of this laboratory study was to evaluate how effective the current filter system at Ohmsett might be in filtering both dispersed oil and dispersant materials. Before discussing the test program, the filter system and procedures at Ohmsett are described.

## 7.1 Filter Operation at Ohmsett

The filtration system at Ohmsett includes a U.S. Filter Co. Autojet 1000 leaf filter that has 28 five-foot diameter septum disks, commonly called "leaves". Each leaf is porous and covered with a nylon mesh. When in use the nylon cloth holds a "pre-coat" which can be one of a variety of materials or combinations of materials, but currently the filter aid used at Ohmsett is diatomaceous earth (DE).

The filter operation is a complex batch process, involving eight steps in each filtration cycle (MAR 1997). Briefly stated, a slurry of DE is introduced into the filter prior to each filtration cycle so that a pre-coat layer of DE is deposited. This takes about 15 minutes. Once the filtering process begins, additional filter aid, called body feed, is added to the filter influent. As the DE-charged filter operates and removes contaminants from the Test Basin, water flow is continuously restricted and reduced, and the differential pressure between the filter inlet and outlet continues to increase. When the differential pressure reaches a certain level or when the operation cycle time reaches 24 hours, the operation is stopped, the filter is cleaned and the process begins again.

## 7.2 Study Approach

The question here is whether the current filter aid, a particular grade of diatomaceous earth, is capable of removing oil and dispersant. A wide variety of DE grades are commercially available. The coarser grades have greater permeability and solids-handling capability than do the finer grades, but the finer grades will generally produce a cleaner effluent. Some grades of DE are pretreated to

improve performance. According to the operations manual for the filter at Ohmsett (MAR 1997), other filter aids are required to sorb finely dispersed oil droplets.

It was recognized at the outset of the study that diatomaceous earth itself might not be adequate to filter fine particles of oil, as suggested in MAR 1997, but this could only be checked by experimentation. This was done, as reported below, and, indeed, the current filter aid proved to be inadequate. So, after some research and discussion with the supplier of filter aids to Ohmsett, a company that supplies a full range of products, another product was found and tested as well. The following is a report on this two-step testing process. For convenience, the test results of both filter aid products are reported together, even though the tests proceeded one after the other.

## 7.3 Test Materials

The oil used in the tests was fresh Alaska North Slope and the dispersant was Corexit 9500. Three kinds of water were used in the tests: filtered Ohmsett water, salt water with salinity of 10 ppt and distilled water. In terms of filter aids tested, the first task was to evaluate the product currently used at Ohmsett. This is a diatomaceous earth product called Celatom Diatomite FW-60, manufactured by Eagle-Picher Minerals, Inc. The other product selected and tested is a ball-milled cellulose fiber product called Celatom Pre-co-Floc PB-100M, also manufactured by Eagle-Picher. A specifications sheet for the Pre-co-Floc product series is provided in Appendix B.

## 7.4 Test Apparatus and Choice of Solvents

The analytical instrument used to measure dispersed oil and dispersant in water was a Horiba OCMA-350 non-dispersive infrared oil content analyzer. This instrument measures the absorption of IR light by a sample in the 3.4 to 3.5 μm wavelength range. This is the wavelength of light absorbed by the C-H bond. In order to measure the oil content of a sample of water, the sample is shaken with a solvent that extracts the hydrocarbons. The solvent used for the extraction and analysis obviously cannot absorb light in the 3.4 to 3.5 μm wavelength range. Up until a few years ago the solvents of choice were carbon tetrachloride and Freon. The use of these solvents is becoming

increasingly restricted because of their ozone-depleting properties. The Horiba OCMA-350 is supplied with a non-banned chloroflurocarbon solvent, S-316, a polymer of chlorotrifluoroethylene. Although this solvent has the necessary property of having no C-H bonds to absorb IR light in the stated wavelength range, its solvating power with crude oil was found to be less than desirable and it is also very expensive (ca. $1000 US for a 4L jug). In any case, this is the solvent that was used to assess the effectiveness of the diatomaceous earth filter aid.

After the tests with DE, it was decided to research alternative solvents. A suitable substitute was found in IR-spectroscopy grade perchlorethylene (also known as tetrachloroethylene). Perchlorethylene has a much better solvating power with crude oil and is much less expensive (ca. $150 US for 4L). It was this solvent that was used to evaluate for the test series with cellulose as the filter aid.

## 7.5 Test Procedure

The tests conducted in this phase were intended to simulate the operation of the Ohmsett leaf filter, but the loadings of filter aid (both the pre-coat and the body-coat) used were higher that those that are used at Ohmsett. The purpose of this was to ensure that any beneficial effects of the filter aids would be easily detected. The tests involved filtering samples of water contaminated with about 50 mg/L of either Alaska North Slope (ANS) crude oil, Corexit 9500 or ANS crude oil pre-mixed with 1:20 Corexit 9500. All sample were vigorously shaken with a wrist-action shaker to ensure thorough dispersion of the contaminant into the water prior to extraction and/or filtering.

The filter used was a ceramic-type Buchner funnel fitted with medium-to-fine Fisher brand P8 filter paper (5.5 cm diameter) and placed on a 1-L glass Kimax filter flask. Vacuum was provided by a water-tap aspirator. Prior to a filter test, a "pre-coat" of filter aid was placed on the filter paper. This involved mixing a slurry of 12 g of Diatomite (or 6 g of Pre-co-Floc) in 200 mL of tap water and filtering the slurry (as a comparison, this is about four-times as much pre-coat as would be normally used on the Ohmsett leaf filter). For a test, 12 g of Diatomite (or 6 g of Pre-co-Floc) was mixed with

the contaminated water and filtered (this is about a 50-times greater concentration of filter aid in the body-coat slurry than would be used with the Ohmsett leaf filter).

## 7.6 Results of Filter Tests

Figure 12 shows the results obtained with the tests involving the diatomaceous earth filter aid, Diatomite FW-60. It is clear that the diatomaceous earth does not remove much Corexit 9500 dissolved in water. Also, it appears that the product only removes about 50% of dispersed oil from water. The figure shows the poor solvating power of the S-316 solvent with crude oil - each of the three ANS-contaminated samples was prepared with a concentration of 50 mg/L. These results led to the conclusion that the diatomaceous earth was not a suitable filter aid for dispersed oil or dissolved dispersant.

Figure 13 shows the much better results obtained with the cellulose filter aid, Pre-co-Floc PB-100. These tests were conducted with distilled water to reduce the problems found in the first test series when extracting salty water with the S-316 solvent. The Pre-co-Floc PB-100M was able to remove virtually all of the dispersed ANS (either crude oil alone or crude oil pre-mixed with 1:20 dispersant). For the oil-in-water samples that were filtered twice (through the same filter cake), additional oil removal was detected in each case.

Although the results with dissolved dispersant were not as spectacular, there is an indication that the cellulose product can remove 50 to 66% of the Corexit 9500 from water as well. As with the S-316 it is noted that the perchlorethylene solvent could not extract all of the dispersant from the water samples - the readings of about 15 mg/L of 9500 on Figure 13 result from extracting 50 mg/L samples. Thus, the readings of approximately 7 mg/L for the filtered water represent something like 23 mg/L (7 x 50 / 15) remaining in the water.

The dispersant removal results are somewhat supported by a series of surface tension measurements made of filtered water. These were:

Surface tension reading for distilled water = 73.6 dynes/cm
Surface tension reading for distilled water containing 50 mg/L of Corexit 9500 = 55.2 dynes/cm
Surface tension reading for filtered water = 61.3 dynes/cm

## 7.7 Additional Bench-Scale Tests at Ohmsett

At this point of the study it was not possible to state definitively that the cellulose would be effective in removing dispersed oil or dissolved dispersant from Ohmsett tank water. Follow-up work was necessary, and was carried out in the lab at Ohmsett. This work had the purpose of determining the best grade and loading of cellulose filter aid. The test plan and results can be found in Appendix D. The conclusions from the tests are summarized below.

1.  The tests conducted in Ottawa were repeated at the Ohmsett lab with two grades of cellulose filter aid (PB-100M and PB-200M) recommended as suitable by the manufacturer. The results indicated no significant difference in performance between the two in removing dispersed oil from Ohmsett tank water. PB-100M was selected for use in the full-scale tests described in the next section because there was more laboratory data on its performance.

2.  Tests with different thicknesses of cellulose on the filter paper indicated that a loading on the Ohmsett leaf filter of 15 to 30 lb/100 ft$^2$ would be appropriate for the full-scale tests.

3.  Several one-hour tests with larger batches of Ohmsett water filtered through a thickness of cellulose filter aid equivalent to a loading of 30 lb/100 ft$^2$ indicated that it would continue to remove dispersed oil droplets over this time frame without significant decline in removal efficiency. Measurements of the surface tension of the filtered water to determine whether or not the cellulose removed dissolved dispersant were not conclusive, but indicated that the filter aid was not an effective removal agent for the dissolved surfactants over the one-hour time span.

# 8. Full-Scale Verification Testing at Ohmsett

## 8.1 Overview

Bench-scale tests were performed first at SL Ross and later at the Ohmsett laboratory to assess the effectiveness of both diatomaceous earth and cellulose in removing dispersed oil and dispersant chemicals from the water in the tank. The tests indicated that the cellulose would remove virtually all dispersed oil, but the results on the removal of dispersant alone were not conclusive: early tests indicated that some dispersant may have been removed by filtering through cellulose but subsequent testing did not confirm this.

The goal of this final task in the study was to verify the findings of the lab-scale work by running a series of tests using the Ohmsett filtering system at full scale.

## 8.2 Background

### 8.2.1 Ohmsett filter system

The filtration system at Ohmsett is described in detail in Chapter 7, section 7.1. Briefly, a slurry of DE is introduced into the filter prior to each filtration cycle so that a pre-coat layer of DE is deposited. This takes about 15 minutes. Until recently, once the filtering process began, additional filter aid, called body feed, was added to the filter influent at specified concentrations. This step is no longer performed due to equipment breakdowns. As the DE-charged filter operates and removes contaminants from the Test Basin, water flow is continuously restricted and reduced, and the differential pressure between the filter inlet and outlet continues to increase. When the differential pressure reaches a certain level or when the operation cycle time reaches 24 hours, the operation is stopped, the filter is cleaned and the process begins again.

Based on the lab-scale testing described in the previous chapter, it was decided to perform the full-scale tests using cellulose rather than DE as the filter aid, and that a pre-coat of 15 lb/100 ft$^2$ would be used.

## 8.3 Methodology of Full-scale Tests

### 8.3.1 Test Set-up

In designing the verification tests described here, it was desirable to operate the filter system at as close to typical design conditions as possible. This meant that, for a one-hour test and a design flow rate of 1800 gpm, as much as 108,000 gallons of water would be passed through the system. This could have been achieved by simply running the filter system in its normal mode of operation and injecting chemically-dispersed oil to the influent upstream of the filter. However, there was uncertainty over whether the filter could remove the dissolved dispersant, and operating the filter system in this manner could lead to a measurable concentration of dispersant in the water in the Ohmsett test tank. Using portable tankage for the required volume of test fluid – influent and effluent – did not seem to be a reasonable solution given that the planned series of tests would require up to 1,000,000 gallons of contaminated fluid. Therefore, the full-scale tests were designed to be carried out with the filter system in a closed loop, comprising the filter and pump, and the piping and fittings connecting the two.

The filter system was isolated from the towing basin by closing valves at the influent and effluent ends of the filter system. This created a closed loop as depicted in Figure 14.
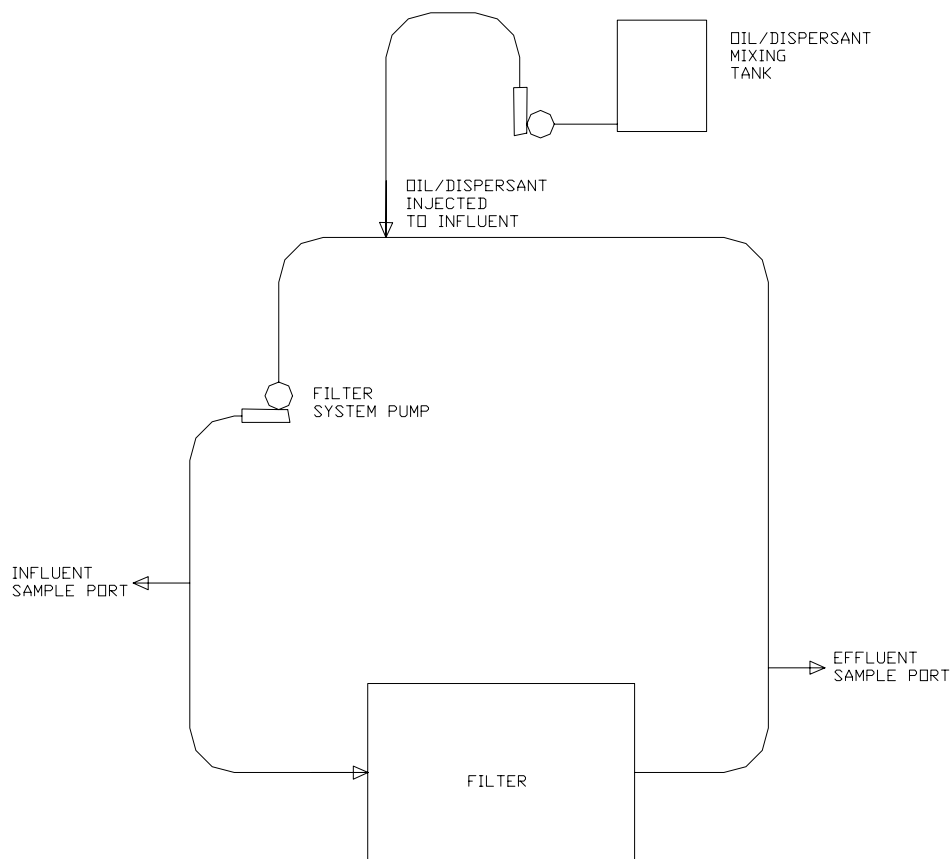
**Figure 14: Schematic diagram of closed-loop test configuration**

Chemically dispersed oil to contaminate the influent water was prepared by first mixing dispersant into a volume of crude oil. For all tests, a dispersant:oil ratio of 1:20 was used. A measured amount of this doped oil was then poured into the now-disused body feed slurry preparation tank containing a pre-determined volume of clean Ohmsett water. While the oil/dispersant mixture was being added and throughout the test, the tank contents were vigorously mixed with a bladed electric mixer to create and maintain small oil droplets in suspension. During each test, a slurry of this dispersed oil

was pumped from the slurry tank into the recirculating water in the closed loop near the chlorinator injection port. Injecting the chemically dispersed oil slurry at this point, just downstream of the filter, allowed the slurry to mix with the recirculating water as it passed through several lengths of pipe, several bends, two strainers and the centrifugal pump. This allowed a thorough blending of the slurry with the recirculating water to achieve the desired influent concentrations.

Influent samples were taken periodically from the pressure gauge valve just upstream of the leaf filter to determine the actual oil-in-water concentration entering the filter. As the influent samples were taken, an effluent sample was also taken from an existing tap in the line just downstream of the filter.

For each test, the water in the test loop was disposed of and replaced with fresh water from the Ohmsett test basin.

Salinity in the Ohmsett test basin was measured to be approximately 15 ppt. There are plans to increase the tank salinity to 35 ppt to better represent offshore conditions. Therefore, at the start of each test, salt was added to the water in the test loop to increase the salinity to approximately 35 ppt. This was accomplished by adding a measured amount of rock salt to water in the pre-coat mixing tank, mixing this vigorously to dissolve the salt, and then pumping this brine mixture into the closed loop while operating the filter in recirculating mode.

**8.3.2 Test Materials**

The oil used in the tests was fresh Alaska North Slope and the dispersant was Corexit 9500. Based on the results of the lab-scale tests, the filter aid selected for use in these full-scale tests was Celatom Pre-co-Floc PB-100M. A specifications sheet for the Pre-co-Floc product series is provided in Appendix B.

**8.3.3 Test Variables**

Based on the suggested dispersant tests and limiting background concentrations of dispersed oil, the filter should be capable of removing oil from the water at influent concentrations on the order of 50 mg/L. This oil-in-water concentration was the main one used in the tests, with additional runs done at a lower and higher concentration -- on the order of 10 and 200 mg/L -- to determine the range of effectiveness.

It was difficult to precisely control the oil-in-water concentration at the proposed levels. In the closed-loop tests, this was partially due to the fact that the filter was not 100% effective and oil that passed through the filter as effluent became included as influent as it cycled through the system. The second problem, which affected both the closed-loop and on-stream tests, was that the pump used to inject the oil/dispersant mixture to the influent stream was difficult to precisely control. As well, since the concentrations in the influent could not be measured in real-time, it was not practical to vary the injection rate to meet a set influent concentration.

Therefore, following test #2, in which a slightly low but acceptable influent concentration of 33 mg/L was achieved, the same influent conditions were used for the remaining tests: the same amount of oil was mixed in the slurry tank and the same influent pumping rate was used. The only exceptions to this were test #4, in which half the volume of oil was used to produce a lower oil-in-water concentration, and in test #8, in which twice the volume of oil was used.

For the first 10 minutes of each test and periodically throughout, the level of the oil/dispersant mixture in the mixing tank was measured to estimate its drawdown and, if necessary, adjustments were made to the rate of the injection pump.

As discussed above, it was planned to use a flow rate at or close to the design flow rate of 1800 gpm throughout the tests. However, due to the air infiltration problems that were experienced during the dry-run testing of the closed loop it was decided to use a lesser flowrate of 1500 gpm. It was felt that this would reduce the rate at which air was admitted to the system and would therefore lengthen the available period for a valid test.

## 8.3.4 Sampling and Analysis

Samples were taken of the filter influent and effluent on a periodic basis to determine filter effectiveness. It was also planned to use this to confirm that the desired influent concentration was being achieved; however, samples could not be analyzed quickly enough to do this in any meaningful way.

The concentration of oil in the water samples was determined using solvent extraction/IR. The concentration of dissolved dispersant in the effluent samples was determined using surface tension measurements. Influent and effluent samples were drawn at five and 10 minutes into each test, and every 10 minutes thereafter.

During the project some interest had developed over whether natural biodegradation could be a means of reducing the dispersant concentrations in the tank. Accordingly, additional effluent samples were taken at the conclusion of tests #2, #3, and #4 to determine whether this was indeed true. For each of these three tests, four effluent samples were drawn and their surface tension measured. Two of the four samples were then stored indoors (in the Ohmsett laboratory) and two were stored outdoors. The surface tension, an indicator of dispersant concentration, was then measured for each sample on a daily basis to determine the reduction, if any, over time.

## 8.3.5 Sequence of Testing

Cellulose is not typically used as the filter aid at Ohmsett, and there was some uncertainty over its handling, flow, pre-coat, sluicing and Oberlin filter cake characteristics. As well, the filter system is not usually run in a recirculating mode except for a 15-minute period while the pre-coat is being applied. Therefore, prior to the start of the tests the filter was run in recirculating mode with uncontaminated water (i.e., from the basin) to gain experience in cellulose use, possible changes in pre-coat and sluicing procedures, and so on.

Following the dry run, the sequence of tests shown in Table 8 was followed. The intention was to run a number of duplicate tests at the main conditions of interest (i.e., 35 mg/L influent concentration and a 15 lb/ft$^2$ filter precoat), with additional runs at a different influent concentrations. As well, three tests were run with double the above precoat loadings to investigate the effect on the removal effectiveness.

**Table 8. Summary of filter test parameters**

| Test # | Oil Influent, mg/L | Precoat, lbs / 100 ft$^2$ | Mode |
|--------|--------------------|---------------------------|------|
| 1 | 35 | 15 | closed loop |
| 2 | 35 | 15 | closed loop |
| 3 | 35 | 15 | closed loop |
| 4 | 5 | 15 | closed loop |
| 5 | 35 | 30 | closed loop |
| 6 | 35 | 30 | on-stream |
| 7 | 35 | 15 | on-stream |
| 8 | 70 | 30 | on-stream |

It was intended to run all of the tests in the closed-loop mode. As discussed later in this chapter, problems with this configuration limited the duration of the test run to about 50 minutes. Further, it was suspected that these problems, inherent to the closed-loop configuration, were negatively affecting the results. Therefore, the final three tests were carried out in an on-stream mode, drawing water from the Ohmsett wave basin, injecting the oil/dispersant mixture to the influent prior to its entering the filter, and discharging the effluent back into the wave basin. It was accepted that this would contaminate the wave basin, but it was felt that this would be acceptable considering that only three tests would be run in this manner.

## 8.4 Results

The following results cover (1) the filter's effectiveness in removing dispersed oil, (2) its effectiveness in removing dispersant, and (3) the natural degradation of dispersant in the effluent.

## 8.4.1 Removal of Dispersed Oil

Of primary concern in these tests was determining the ability of the Ohmsett filter system to remove dispersed oil from the water. If the filter were effective it would be possible to reduce the oil-in-water concentrations in the tank to acceptable levels and thus be able to carry out a series of dispersant tests over a period of a week or more.

The entire results, showing the influent and effluent oil concentrations for the eight tests are presented in Appendix C. These are summarized in Figures 15 through 17 and in Table 9 below. Figures 15 and 16 show the results for the first five tests which were done with the filter in a closed-loop. Each graph shows the influent and effluent concentration at various times through the test run and the calculated reduction in oil concentration (i.e., influent minus effluent, divided by influent). Note that the system was allowed to continue for up to 15 minutes after the oil injection was stopped, generally at around the 50-minute mark. This accounts for the sharp drop in the influent oil concentration for the last two or three data points on each graph; removal efficiency is not calculated for these data. In each of these closed-loop tests, there is a gradual decrease in removal efficiency through the 50-minute test period. Initial removal efficiencies are in the range of 60 to 80%, but by the end of each test, these have dropped to 20% or lower.

A likely explanation lies in the problems inherent to the closed-loop test setup. Running the filter in a closed-loop was problematic in that air gradually built up in the loop and eventually caused the pump driving the system to lose prime. As a result, the water level within the filter housing gradually declined. This meant that the upper portion of the filter leaves were exposed to a turbulent mixture of air and the oil/water influent, rather than being wetted and under suction pressure. It was suspected that this caused some of the cellulose to fall off the filter leaves, which would allow the oil/water influent to pass through unfiltered. The filter housing was opened after several of the closed-loop test runs to determine if this were so.

Figure 18 shows the opened filter housing following test #5. While perhaps not evident in this photograph, areas near the top of the filter leaf were not caked with cellulose as they should have been. Note that the bottom third of the filter leaf is also bare of cellulose. This occurred during the shutdown of the system and the rapid draining of the filter housing. It is clear that the bare areas near the top of the filter leaf lost their cellulose during the test; this was evidenced by the coating of oil directly on the nylon filter which is not seen on the lower third of the filter.

**Figure 15:** Removal of dispersed oil (Tests # 1 to 3)
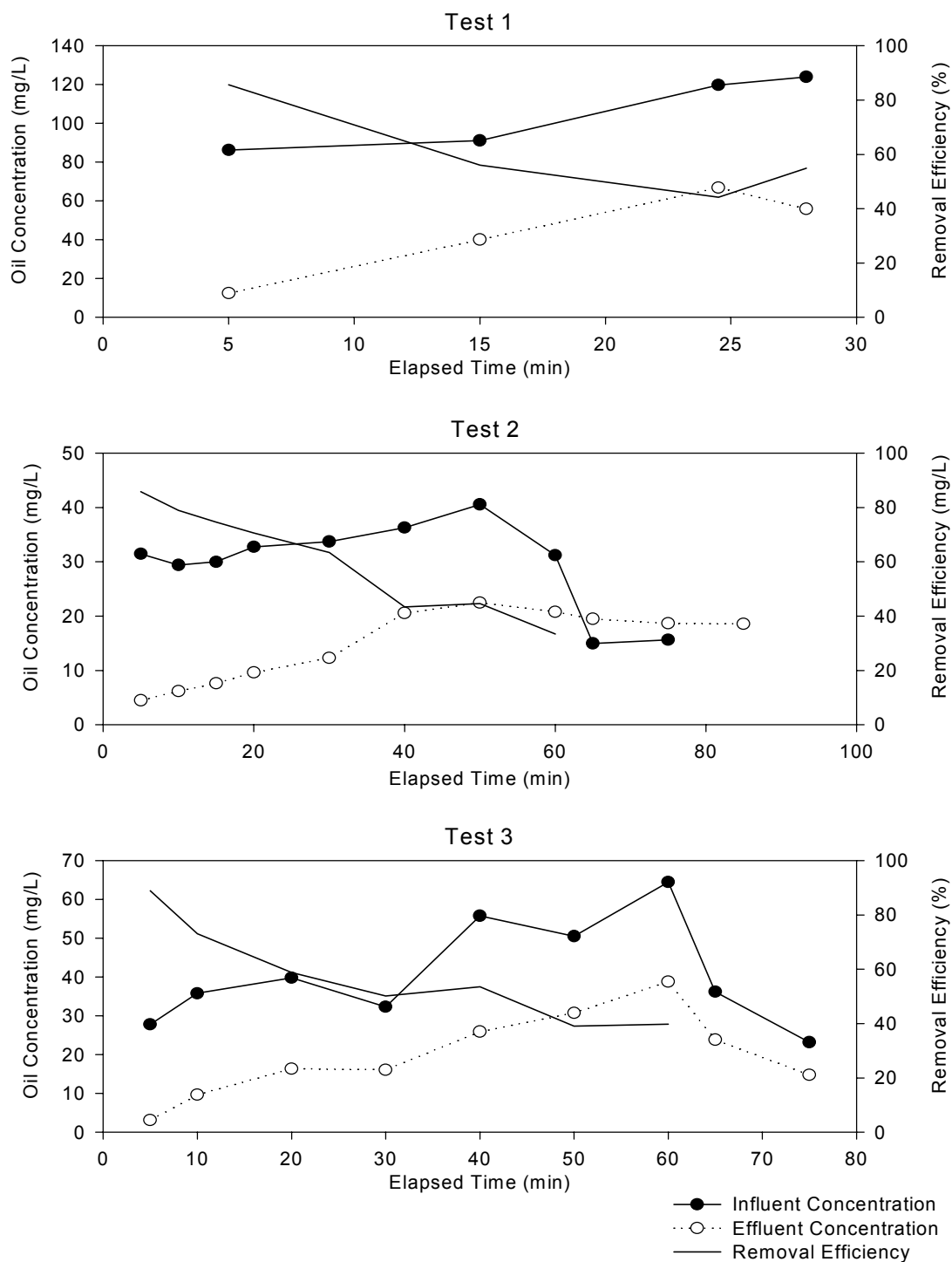
**Figure 16:** Removal of dispersed oil (Tests # 4,5)
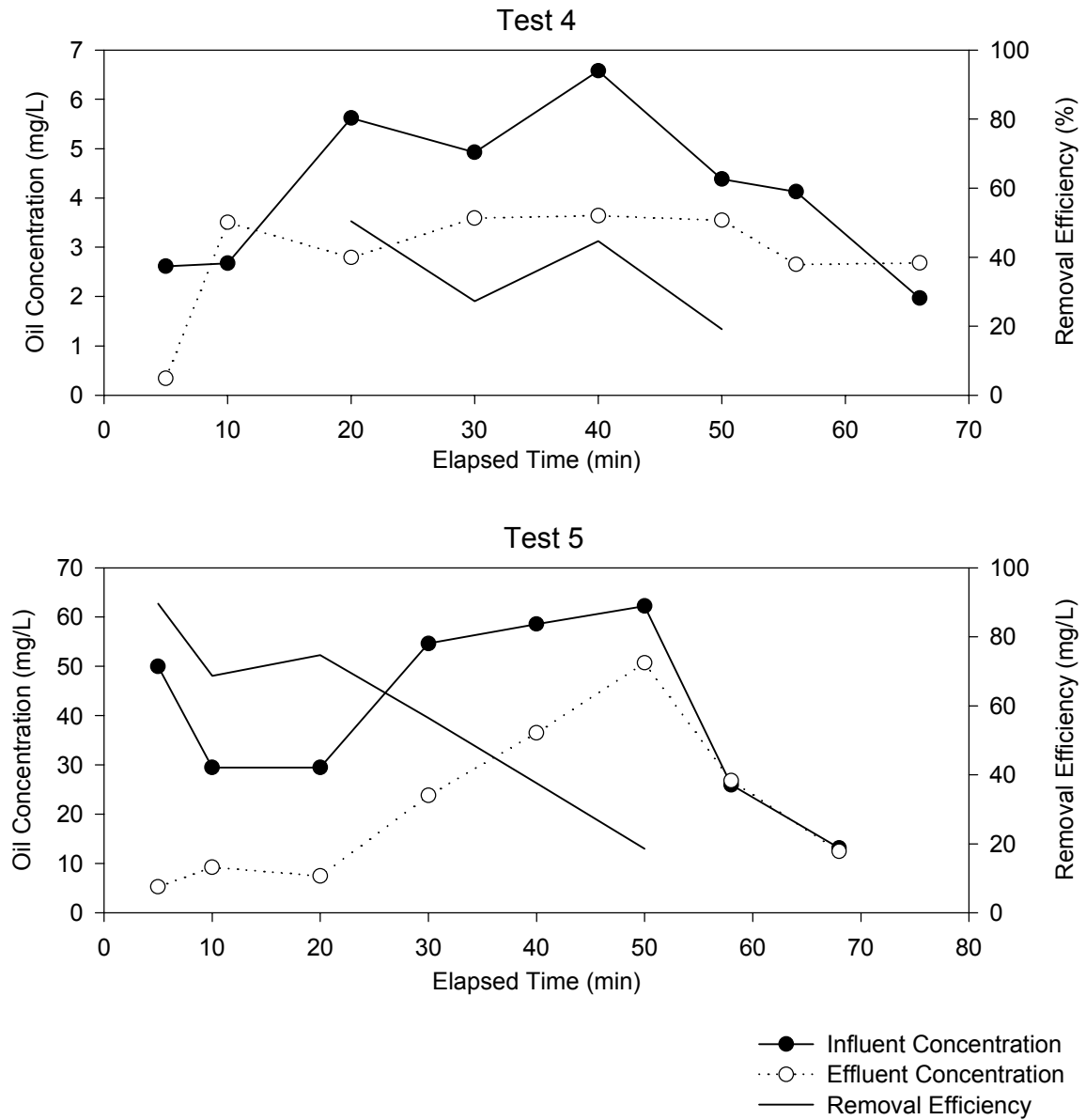
**Figure 17:** Removal of dispersed oil (Tests # 6 to 8)
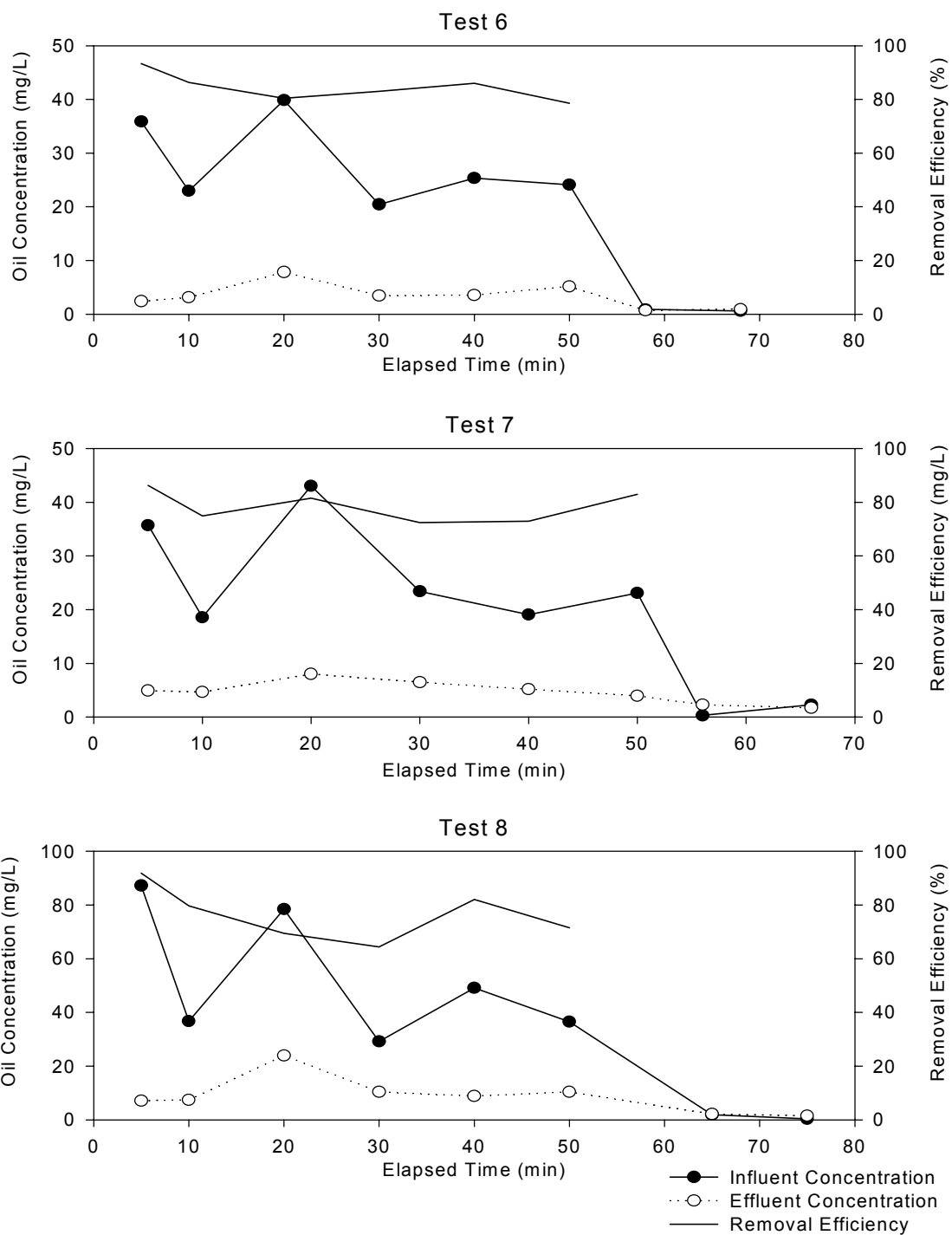
**Figure 18: View of filter leaf following closed-loop test #5**

Figure 17 shows the results of the "on-stream" tests, #6 through 8. In these tests, more consistent removal efficiencies were obtained. The removal efficiencies ranged from 64 to 93% and averaged 81%. Unlike the results of the closed-loop tests, the removal efficiencies did not exhibit a downward trend over the test period. Examining the average removal efficiencies, shown in Table 9, there is no significant difference in the results for the three tests which covered "standard" conditions of 30 mg/L influent and 15 lbs / 100 ft$^2$ precoat (test #7), same influent with double the precoat (#6), and double the influent concentration and double the precoat.

**Table 9. Summary of results - removal of dispersed oil**

| Test # | Precoat, lbs / 100 ft$^2$ | Avg. influent conc., mg/L | Avg. effluent conc., mg/L | Removal Efficiency, % |
|---|---|---|---|---|
| Closed-loop tests | | | | |
| 1 | 15 | 105 | 44 | 58 |
| 2 | 15 | 33 | 13 | 61 |
| 3 | 15 | 44 | 20 | 54 |
| 4 | 15 | 4.5 | 2.9 | 35 |
| 5 | 30 | 47 | 22 | 53 |
| average | | | | 52 |
| On-stream tests | | | | |
| 6 | 30 | 28 | 4.3 | 85 |
| 7 | 15 | 27 | 5.5 | 80 |
| 8 | 30 | 53 | 11 | 79 |
| average | | | | 81 |

## 8.4.2 Removal of Dispersant

A second concern in these tests was whether or not the cellulose would effectively remove dispersant from the influent water. The lab-scale results were ambiguous, but it was suspected that there would be a limited effectiveness if any.

Using the same samples as for the dispersed oil analysis, the surface tension was measured for the influent and effluent. Prior to doing the tests, a calibration curve was prepared relating the dispersant-in-water concentration to the surface tension (see Appendix D). The results of the tests are shown in Table 10.

**Table 10. Summary of results - removal of dispersant**

| Test # | Avg. influent oil conc., mg/L | Avg. dispersant influent conc.[1], mg/L | Avg. dispersant effluent conc.[1], mg/L |
|--------|-------------------------------|------------------------------------------|------------------------------------------|
| Closed-loop tests | | | |
| 1 | 105 | 16 | 14 |
| 2 | 33 | 22 | 16 |
| 3 | 44 | 22 | 16 |
| 4 | 4.5 | 4.5 | 2.4 |
| 5 | 47 | 18 | 22 |
| On-stream tests | | | |
| 6 | 28 | 14 | 7.3[2] |
| 7 | 27 | 11 | 1.4 |
| 8 | 53 | 9.5 | 3.5 |
| [1] - dispersant concentration calculated based on measured surface tension | | | |
| [2] - average due mainly to one anomalous data point, without which average would be 1.8 mg/L | | | |

Focusing on the results for the "on-stream" tests, at first glance it appears that the filter does indeed remove an appreciable amount of the dispersant. However, the data only points out the flaw in the

use of surface tension as an indicator for dispersant concentration. While it is certainly valid as an indicator for indicating the concentration of dispersant in clean water, or perhaps water with a low oil concentration, it appears to be unsuitable for indicating dispersant concentration in water with oil concentrations in the range of the influent concentration here. For example, in test #7, the influent had an average oil concentration of 27 mg/L. Based on the dispersant dosage of 1:20, the influent should have had a concentration of approximately 1.35 mg/L (i.e., $27 \div 20$). The calculated value of 11 mg/L, based on the measured surface tension, is clearly erroneous. On the other hand, this value of 1.35 is close to the calculated concentration for the effluent concentration, and a similar comparison can be made with the effluent concentrations for tests #6 and #8. The conclusion is that the filter had not removed the dispersant.

### 8.4.3 Natural Degradation of Dispersant from Effluent Samples

Samples of effluent were taken after three of the tests to determine if the dispersant remaining in the water would break down over time due to natural degradation. The surface tension was measured for these samples over a two-week period. The measured surface tensions, and the inferred dispersant concentrations, are detailed in Appendix E. For the samples collected after tests #2 and #3, no significant change was measured in the surface tension over the two-week period. For the effluent samples collected after test #4, the surface tension did increase from about 50 mN/m to about 70 mN/m. This indicates a final dispersant concentration, after the two-week period, of between 1.1 and 1.7 mg/L which, as noted in the previous discussion, is close to what one would expect in the influent based on the 1:20 ratio of dispersant in the oil injected into the influent. It was concluded that natural degradation had no measurable effect on the dispersant concentration in the effluent.

## 8.5 Conclusions

A suspected leak in the closed loop allowed air to enter the system during the first five tests using the closed-loop mode. This air created turbulence within the filter which may have loosened cellulose from the filter leaves and reduced its effectiveness. Further, the build-up of air in the system limited the period available for a test run to about 50 minutes, after which time the pump

driving the system began to lose prime. This was a problem related solely to running the system in a closed loop for the tests and should not present a problem in actual running of the filter system in its normal mode of operation. In fact, in subsequent tests with the filter "on-stream" in its normal operating mode, no such problem occurred.

Using cellulose as the filter aid as opposed to diatomaceous earth presented no particular difficulties in handling, slurry preparation, or in the pre-coating procedure.

Cellulose appears to be effective in removing dispersed oil from the Ohmsett tank water. In the initial closed-loop tests using a pre-coat loading of 15 lb/100 ft$^2$, an average of 52% of dispersed oil was removed from an influent stream with oil-in-water concentrations ranging from 5 to 105 mg/L. In each of the closed-loop tests, the effectiveness of the filter generally declined through the one-hour test period

In the on-stream tests, which would be more representative of typical filter operation following a dispersant test, an average of 81% of the dispersed oil was removed from an influent stream that had an oil-in-water concentration ranging from 23 to 87 mg/L. In the on-stream tests, the filter effectiveness varied less through the test period.

Cellulose does not appear to be effective in removing dispersant from the water. Measurements of the surface tension of the effluent water indicate dispersant concentrations of at least 1 to 2 mg/L, which corresponds to the influent concentration of dispersant calculated by a mass balance. This will not limit the ability to carry out a series of dispersant tests, but following those tests, treatment of the tank water will be required prior to containment boom or skimmer testing.

Tests to determine whether natural degradation could significantly break down the remaining dispersant in the effluent water indicated that there likely was no measurable degradation over a two-week period.

# 9. Bench-Scale Testing of Activated Carbon

As a result of the bench-scale tests and subsequent full-scale tests at Ohmsett, it was concluded that dispersed oil could be removed from Ohmsett tank water using the existing filter system with cellulose as the filter aid, but that the existing system was inadequate for removing dissolved dispersant from the water. It was decided to do a small series of tests with activated carbon to determine its efficacy to remove dispersant.

A series of bench-scale tests were performed in the Ohmsett lab following the procedure outlined in ASTM D 3860-98. Briefly, samples containing a known concentration of contaminant (i.e., dispersant in this case) are prepared, various measured amounts of pulverized activated carbon are added to the samples, and after a contact period of two hours, the mixture is filtered through a specified membrane filter. The results are reported as the amount of contaminant adsorbed per unit weight of the carbon.

Table 11 summarizes the results of the bench-scale testing. The results clearly indicate that activated carbon will remove dispersant from Ohmsett tank water. Comparing the influent to effluent concentrations of dispersant it is seen that 90 to 100% of the Corexit is removed. Further testing is required with lower volumes of activated carbon to determine the optimum volume of carbon, and to allow a determination of the amount required for filtering Ohmsett water over a range of dispersant concentrations.

**Table 11. Summary of results - removal of dispersant using activated carbon**

| Corexit conc., mg/L | Carbon weight, g | Surface tension, dynes/cm | | Corexit remaining, mg/L | Corexit adsorbed per unit carbon, mg/L |
|---|---|---|---|---|---|
| | | pre-filter | post-filter[1] | | |
| 10 | 0.01 | 48 | 70.7 | 1 | 90 |
| 10 | 0.02 | 45.5 | 70.8 | 1 | 45 |
| 10 | 0.04 | 44 | 73.7 | 0 | 25 |
| 10 | 0.1 | 47 | 71.0 | 1 | 9 |
| 10 | 0.2 | 45 | 71.0 | 1 | 4.5 |
| 20 | 0.01 | 10 | 74.7 | 0 | 200 |
| 20 | 0.02 | 40 | 73.3 | 0 | 100 |
| Notes: All tests carried out with water salinity of 35 ppt. Post-filter surface tension is average of three replicates. | | | | | |

# 10. Conclusions

1.  Dispersant testing at Ohmsett offers two distinct advantages over testing at sea: (1) the ability to alter and control variables in a systematic way; and (2) the much lower costs of the testing. These are the same advantages that have justified the activity at Ohmsett in the area of boom and skimmer testing.

2.  Laboratory tests indicate that the presence of dispersant in Ohmsett water following dispersant tests will strongly affect the interfacial tension of oils used in subsequent boom or skimmer tests at Ohmsett. Even concentrations as low as a few ppm may be enough to cause dramatic effects. Therefore it must be assumed that even small concentrations of dispersant in the water are unacceptable for conventional equipment testing at Ohmsett, and that the tank will have be thoroughly cleaned of dispersant following a dispersant testing program.

3.  The results of dispersant effectiveness testing, with dispersant added to the water rather than to the oil, show that dispersant concentrations in the water can reach at least 400 ppm before having a noticeable effect on the dispersion of floating oil. This means that many dispersant experiments could be done consecutively, with increasing dispersant concentrations in the tank, without concern that the dispersant in the tank were having an effect on the dispersant effectiveness results.

4.  Turbidity studies and analysis indicate that underwater oil behavior effects from dispersant testing at Ohmsett will only be visible by keeping turbidity to very low levels. If dispersed oil concentration in the tank reaches a value of only a few ppm, it will be impossible to view underwater effects. Because each dispersant experiment will certainly lead to these concentrations and higher, only the first experiment in a series of dispersant experiments will be visible underwater and be recordable by video or photography.

5. An analysis scoping out the experimental possibilities of testing dispersants at Ohmsett suggests that a number of dispersant experiments could be run in sequence at Ohmsett before having to filter the body of water to remove dispersed oil. Perhaps two to four experiments could be run in a row involving about 26 to 53 gallons of oil each.

6. A series of bench-scale tests followed by a full-scale test using the existing filter system at Ohmsett showed that it is possible to remove dispersed oil from Ohmsett tank water. An average of 81% of the dispersed oil was removed using cellulose as the filter aid.

7. It was found that cellulose was not effective in removing dissolved dispersant from the water. A preliminary series of bench-scale tests were performed and these indicate that activated carbon will remove dissolved dispersant with a high degree of effectiveness. Further testing with activated carbon will be required to determine the amounts of material required for a range of dispersant concentrations and tank-water volumes.

8. Overall, the study concludes that there are several restraints to dispersant testing at Ohmsett, but that such testing could be done with good success if the testing program is carefully designed and implemented with due regard for the limitations noted above.

# 11. References

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**Appendix A:**

**WSL Laboratory Dispersant Test**

**(from Nordvik et al. 1993)**

# The Determination Of The Efficiency Of Oil Spill Dispersants

## INTRODUCTION

The method for the determination of the Efficiency Index of oil spill dispersants is defined in the report LR448 (OP). This document expands upon the analytical procedures used in following the above method, it does not change that method.

## DEFINITION

The Efficiency Index of an oil spill dispersant is defined as the percentage by weight of the test oil which has been transferred as small droplets into the water phase under the conditions of the test, assuming complete and even distribution at the time of sampling.

## SUMMARY OF THE METHOD

The oil spill dispersant is added, dropwise to a measured volume of the test oil on the surface of seawater at 10°C in a conical separating funnel.

The separating funnel is rotated about a horizontal axis at right angles to its longitudinal axis, for a period of 2 minutes at 33 ±1 rpm. After rotation has ceased, the stopper is removed and after 1 minute standing time, 50 ml of oily water are run off through the bottom tap. The quantity of oil present in the sample is then determined spectrophotometrically after extraction into chloroform.

The method may be used for determining the efficiency index of any of the three dispersant types (Types 1, 2 or 3).

## APPARATUS

### Separating funnels
(1) A conical separating funnel of nominal capacity 250 ml. After use the funnel should be rinsed thoroughly with chloroform to remove any oil remaining inside, then washed in the washing up machine. The washing program should include a thorough rinse with distilled water to ensure that all traces of detergent are removed. The funnel should then be allowed to drain and cool before being placed in the temperature controlled cabinet ready for use.

(2) A separating funnel to BS2021 with a nominal capacity of 100 ml.

### Motor driven rack
A motor driven rack into which can be fitted and clamped the 250 ml separating funnel. The rack should rotate about a horizontal axis approximately 15-20 mm below the level of the seawater in the separating funnel.

<u>Syringes</u>
(1) A glass syringe fitted with a needle, capable of accurately dispensing 0.2 ml of dispersant in drops of 5 to 10 microlitres.

(2) A glass syringe capable of accurately dispensing 5.0 ml of the test oil.

(3) A glass syringe fitted with a needle, capable of accurately dispensing 2.0 ml of Type 1 dispersant and 2.0 ml of pre-diluted Type 2 dispersant in drops of 5 to 10 microlitres.

<u>Spectrophotometer</u>
A spectophotometer capable of measuring absorbance at 580 nm, and equipped with glass cells of 10 mm path length.

## REAGENTS

<u>Test Oils</u>
(1) An oil of known density and having the following characteristics:

> Dynamic viscosity at 10°C :  1800 to 2200 mPa s at 4 s⁻¹ shear
> Asphaltenes (IP143/78):  6.0% by weight maximum
> Pour point (IP15/67):  below 5°C

(2) An oil of known density having a Dynamic viscosity at 10°C  of 450 to 550 mPa·s at a shear rate of 4 s⁻¹.  This oil is prepared by diluting the higher viscosity oil, referred to above, with kerosine to reduce its viscosity to the required value.

<u>Kerosine</u>
Odourless kerosine to BS 2869 Ams 1 and 2, Class C1

<u>Sea Water</u>
Sea water taken from the sea and having a total solids content of 3.3 - 3.5%.
Synthetic sea water is also permissible.

<u>Chloroform</u>
Sodium sulphate anhydrous.

## METHOD

<u>Calibration</u>
Transfer 0.1, 0.2, 0.3, 0.4, and 0.5 g of the test oil, accurately weighed, into separate 100 ml volumetric flasks.  Dissolve the oil with chloroform and make each flask up to the mark.

Measure the absorbance of each solution at 580 nm in glass cells of 10 mm path length, using chloroform as the reference solution.

A graph may be plotted of absorbance against concentration of oil in the chloroform solutions. Alternatively, using a programmable calculator calculate the line of best fit using a linear

regression programme. The calculator may then be used to covert the sample absorbencies to concentrations.

It is advisable, always to plot the points on a graph to check that no rogue points are included in the least squares regression calculation.

Procedure

The test procedure is carries out in a temperature controlled cabinet, maintained at 10°C . All reagents and test materials should be kept in this cabinet for 24 hours before conducting the test in order to equilibrate them to 10°C .

Place the unstoppered 250 ml separating funnel in position in the motor driven rack. In a measuring cylinder, measure 250 ml of sea water at 10°C and transfer to the separating funnel.

Using the syringe, transfer 5 ml by weight of the test oil to the surface of the sea water and start the stopclock. Weigh the syringe before and after use to calculate the weight (± 0.001 g) of 5 ml of the oil.

Using the appropriate syringe, take the required volume of the dispersant to be tested, (0.2 ml of Type 3 dispersants, 2.0 ml of undiluted Type 1 dispersants and 2.0 ml of freshly diluted Type 2 dispersants). 1 minute after completing the addition of the test oil to the sea water, transfer the dispersant to the oil. The addition of the dispersant to the oil should be done dropwise, starting from the centre of the oil lens and working radially outwards so that the dispersant is distributed as evenly as possible. (See Note 1). Place the stopper in the separating funnel and clip on the retaining cap of the motor driven rack. Close the door of the temperature controlled cabinet.

When the time on the stopclock shows 2.5 minutes from the addition of the oil to the sea water, start the rotation of the separating funnel, and continue for 2 minutes.

When the rotation ceases, allow the funnel to stand undisturbed for exactly 1 minute before running off 50 ml of the oily water into a 50 ml measuring cylinder. The taking of this sample should take no longer than 10 seconds.

The remainder of the determination may be done outside the temperature controlled cabinet.

Transfer the 50 ml sample from the measuring cylinder to a 100 ml separating funnel. Wash the measuring cylinder twice, with 10 ml of chloroform and add these washings to the 100 ml separating funnel. Stopper the funnel and shake for 1 minute. Allow the phases to separate completely and run off the chloroform layer into a 100 ml volumetric flask through a 75 mm glass filter funnel fitted with a 9.0 cm Whatman No. 1 filter paper into which has been placed no more than 1.5 g of anhydrous sodium sulphate. Repeat the chloroform extraction twice more using 20 ml chloroform on each occasion. Thoroughly wash the filter paper and sodium sulphate with chloroform and then dilute the volumetric flask to the mark, stopper and shake well.

Measure the absorbance of the sample solutions using 1 cm cells and a spectrophotometer set to a wavelength of 580 nm. The calibration standards prepared from the test oil being used,

should be measured at the same time, and the calibration line of best fit calculated. A graph should also be plotted in order to check that there are no rogue data points. Using the calculated line of best fit, convert the absorbance readings from the sample solutions into concentrations expressed as, "grams of oil per 100 ml of chloroform". This is equivalent to the weight of oil contained in the 50 ml sample of oily water taken from the 250 ml separating funnel.

Calculation
Calculate the Efficiency Index as follows:

% Efficiency = weight of oil in 50 ml sample of oily water x 500
                weight of oil added to the 250 ml separating funnel

Report
The Efficiency Index is the average of three separate determinations. The calculated average is reported to one decimal place for each of the two reference fuel oils.

**Appendix B:**

**Commercial Literature on Celatom Filter Aids**

# **EPM**

# *Celatom* Filter Aids
by EAGLE-PICHER MINERALS, INC.

# *Pre-co-Floc* Cellulose Fiber

## Performance

Cut Fiber - Soft Wood
(PB-33)
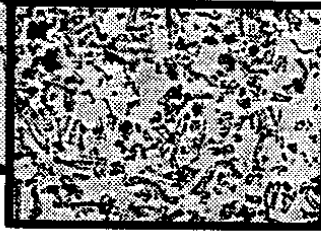
Ball Milled Fiber - Hard Wood
(PB-100M)

### • Cellulose Fiber •

*Pre-co-Floc* cellulose fiber filter aids are derived from purified virgin wood pulp and are 99.9% pure. The sulfite process used in the production of the bleached grades yields a filter aid of exceptional purity (i.e. very low water and/or acid solubles). Cellulose produces almost no ash, is essentially nonabrasive, and is relatively inert to acids, alkalis and solvents. These products are preferred precoat media for processes which are sensitive to silica. Cut fibers are relatively long and have the highest permeability and bridging capabilities. They are made from soft and hard woods. Ball milled fibers are from hard woods, are shorter in fiber length and have the ability to filter out finer solids.

## *Application*

The typical use for cellulose fiber is as a (1) precoat media, (2) an admix to a precoat media and/or as a (3) remedial precoat in a dual precoat process. Most applications are on pressure filters (and some rotary vacuum precoat filters) with wire mesh screens. Proper filter cloth design should obviate the necessity for cellulose precoat application with a natural or synthetic woven media septum. Cellulose fiber is not typically used as a bodyfeed material.

Pressure
Filters
• vertical leaf
• horizontal leaf
• tube

## *Benefits*

- • Prevents Fine Particulate Bleed-Through
- • Enhances Filtered Cake Solids Release
- • Improves Precoat Cake Stability
- • Remedial Precoat For Torn/Worn Septum
- • Offers No Silica Contamination
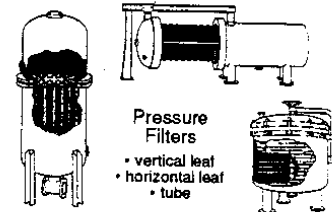
## *Usage Rates*

An effective loading rate ratio for cellulose fiber used as a precoat or as a remedial precoat is 5-7 pounds per 100 square feet of filtration area. The filter aid should be mixed in clean precoat liquor and allowed to wet for at least 15 minutes before being applied to the filter.

For admix applications with other Celatom filter aids, 3-4 pounds per 100 square feet of filter area is satisfactory. It may be more convenient to use Dialose, a premixed blend of diatomaceous earth and cellulose fiber (details on back).

|  | | Cut Fiber - Soft Wood | | | | | Hard Wood | Ball Milled Fiber - Hard Wood | | |
|  | | Unbleached | | Bleached | | | | | | |
| Grades: | | P-30 | P-32 | PB-20 | NB-10 | PB-33 | PB-40 | PB-40M | PB-100M | PB-200M |
| Dry Bulk Density, (lb/ft3) | | 6.0 | 7.0 | 5.0 | 5.0 | 7.0 | 6.0 | 8.5 | 14.0 | 15.5 |
| Wet Bulk Density, (lb/ft3) | | 11.0 | 13.0 | 8.5 | 8.0 | 10.0 | 9.5 | 13.0 | 17.0 | 19.0 |
| Specific Gravity | | 1.58 | 1.58 | 1.58 | 1.58 | 1.58 | 1.58 | 1.58 | 1.58 | 1.58 |
| pH (10% slurry) | | 5.7 | 7.5 | 7.0 | 6.5 | 7.0 | 7.0 | 5.5 | 5.5 | 5.5 |
| G.E. Brightness | | 25 | 25 | 91 | 95 | 92 | 93 | 92 | 90 | 89 |
| Water Absorption (% by weight) | | 370 | 300 | 450 | 420 | 400 | 385 | 315 | 250 | 235 |
| Oil Absorption (% by weight) | | 190 | 165 | 350 | 270 | 250 | 220 | 190 | 130 | 125 |
| Moisture (%) | | 6.0 | 6.0 | 5.5 | 2.5 | 6.0 | 6.0 | 6.0 | 6.0 | 6.0 |
| Ash (% @ 260°C) | | 1.30 | 1.30 | 0.16 | 0.13 | 0.16 | 0.30 | 0.15 | 0.15 | 0.15 |
| Water Solubles (%) | | 2.28 | 2.61 | 0.37 | 0.36 | 0.72 | 0.65 | 0.40 | 0.62 | 0.41 |
| Acid Solubles (% N/10 HCl) | | 2.84 | 2.54 | 0.43 | 0.31 | 0.82 | 0.61 | 0.51 | 0.95 | 0.57 |

**Chemical Analysis Acid Soluable - N/10 HCl  (values reported as %)**

| | | P-30 | P-32 | PB-20 | NB-10 | PB-33 | PB-40 | PB-40M | PB-100M | PB-200M |
|---|---|---|---|---|---|---|---|---|---|---|
| Aluminum Oxide | $Al_2O_3$ | 0.042 | 0.023 | 0.0007 | 0.006 | 0.004 | 0.0002 | 0.0002 | 0.0001 | 0.0004 |
| Iron Oxide | $Fe_2O_3$ | 0.0040 | 0.0035 | 0.000021 | 0.00002 | 0.00012 | 0.00037 | 0.00106 | 0.00111 | 0.00247 |
| Magnesium Oxide | $MgO$ | 0.0247 | 0.0162 | 0.0043 | 0.0044 | 0.0177 | 0.0103 | 0.0081 | 0.0117 | 0.0081 |
| Calcium Oxide | $CaO$ | 0.101 | 0.053 | 0.004 | 0.008 | 0.050 | 0.007 | 0.016 | 0.038 | 0.022 |
| Sodium Oxide | $Na_2O$ | 0.615 | 0.471 | 0.102 | 0.060 | 0.174 | 0.106 | 0.101 | 0.204 | 0.062 |
| Potassium Oxide | $K_2O$ | 0.023 | 0.018 | 0.0044 | 0.0008 | 0.007 | 0.004 | 0.002 | 0.003 | 0.002 |

**Typical Screen Analysis**

| | | P-30 | P-32 | PB-20 | NB-10 | PB-33 | PB-40 | PB-40M | PB-100M | PB-200M |
|---|---|---|---|---|---|---|---|---|---|---|
| +40 Mesh (% on 40 mesh) | | 3.0 | trace | 6.0 | 0.5 | 0.5 | trace | 1.5 | 0.0 | 0.0 |
| -100 Mesh (% through 100 mesh) | | 75.0 | 93.0 | 60.0 | 85.0 | 87.0 | 90.0 | 93.0 | 95.0 | 96.0 |
| -200 Mesh (% through 200 mesh) | | 30.0 | 47.0 | 23.0 | 30.0 | 33.0 | 40.0 | 57.0 | 73.0 | 83.0 |
| Average Fiber Length (microns) | | 75 | 70 | 200 | 110 | 125 | 100 | 50 | 40 | 30 |
| Average Fiber Width (microns) | | 10-40 | 10-40 | 10-40 | 10-40 | 10-40 | 10-40 | 10-40 | 10-40 | 10-40 |

*Dialose*™   A Packaged Blend of *Celatom* Diatomaceous Earth & *Pre-co-Floc* Cellulose Fiber Filter Aids

Product Specifications & Designations

| Available Diatomaceous Earth Filter Aid Grades | Available Cellulose Fiber | *Dialose* Designation | % Cellulose Fiber = (X) |
|---|---|---|---|
| FP-4 | | 4 - X | A = 5% |
| FW-6 | | 6 - X | B = 7-1/2% |
| FW-12 | PB-40M | 12 - X | C = 10% |
| FW-20 | | 20 - X | D = 15% |
| FW-60 | | 60 - X | E = 20% |

• Example •

*Dialose* 6-C
FW-6 with 10% cellulose

Convenient to Use • Consistent Blend • Accurate % Mixture • Reduced Inventory Requirements

CERTIFIED
ISO 9002

(NSF)

EPM

L-100CL

**Appendix C:**

**Detailed Results of**

**Full-scale Tests at Ohmsett**

W34 Filter Test
10/18/99-10/22/99

| IR log# | Sample ID | Surface Tension | Sample Volume | Extraction Volume | Extraction/Volume Extr/Smp | TPH | measured TPH | actual TPH | TPH removal efficiency | Salinity |
|---|---|---|---|---|---|---|---|---|---|---|
| 070 | 1-I-1 | 51.5 | 460 | 100 | 0.217 | 336.60 | 73.2 | 86.2 | | 35.0 |
| 071 | 1-E-1 | 57.5 | 380 | 50 | 0.132 | 90.20 | 11.9 | 12.5 | 85.6% | |
| 072 | 1-I-2 | 48 | 470 | 100 | 0.213 | 363.00 | 77.2 | 91.1 | | AVG INFL |
| 073 | 1-E-2 | 48 | 350 | 50 | 0.143 | 243.60 | 34.8 | 40.0 | 56.0% | 105.2247 |
| 074 | 1-I-3 | 37 | 500 | 100 | 0.200 | 505.40 | 101.1 | 119.7 | | AVG EFFL |
| 075 | 1-E-3 | 64 | 320 | 50 | 0.156 | 365.30 | 57.1 | 66.8 | 44.2% | 43.78512 |
| 076 | 1-I-4 | 48 | 450 | 100 | 0.222 | 470.40 | 104.5 | 123.9 | | REDUCTION |
| 077 | 1-E-4 | 44 | 390 | 50 | 0.128 | 373.90 | 47.9 | 55.8 | 54.9% | 58.39% |
| | | | | | | | | | | |
| 078 | 2-Control | 72.5 | 470 | 50 | 0.106 | 23.13 | 2.5 | 1.1 | | 39.5 |
| 082 | 2-I-1 | 52 | 480 | 100 | 0.208 | 132.80 | 27.7 | 31.5 | | |
| 083 | 2-E-1 | 69.5 | 330 | 50 | 0.152 | 34.50 | 5.2 | 4.5 | 85.8% | |
| 084 | 2-I-2 | 52 | 500 | 50 | 0.100 | 259.60 | 26.0 | 29.4 | | AVG INFL |
| 085 | 2-E-2 | 60.5 | 430 | 50 | 0.116 | 57.30 | 6.7 | 6.2 | 78.9% | 33.16063 |
| 086 | 2-I-3 | 50 | 500 | 50 | 0.100 | 264.40 | 26.4 | 30.0 | | |
| 087 | 2-E-3 | 60 | 440 | 50 | 0.114 | 69.00 | 7.8 | 7.6 | 74.6% | AVG EFFL |
| 088 | 2-I-4 | 52.5 | 500 | 50 | 0.100 | 287.30 | 28.7 | 32.7 | | 13.00189 |
| 089 | 2-E-4 | 58 | 450 | 50 | 0.111 | 85.60 | 9.5 | 9.6 | 70.6% | |
| 090 | 2-I-5 | 50.5 | 490 | 50 | 0.102 | 289.40 | 29.5 | 33.7 | | REDUCTION |
| 091 | 2-E-5 | 49.5 | 450 | 50 | 0.111 | 105.80 | 11.8 | 12.3 | 63.4% | 60.79% |
| 092 | 2-I-6 | 43 | 500 | 50 | 0.100 | 316.80 | 31.7 | 36.3 | | |
| 093 | 2-E-6 | 42.5 | 400 | 50 | 0.125 | 148.81 | 18.6 | 20.5 | 43.4% | |
| 094 | 2-I-7 | 42 | 500 | 50 | 0.100 | 352.20 | 35.2 | 40.5 | | |
| 095 | 2-E-7 | 43.5 | 460 | 50 | 0.109 | 185.80 | 20.2 | 22.5 | 44.6% | |
| 096 | 2-I-8 | 41 | 450 | 50 | 0.111 | 247.20 | 27.5 | 31.2 | | |
| 097 | 2-E-8 | 43 | 420 | 25 | 0.060 | 316.00 | 18.8 | 20.8 | 33.4% | |
| 098 | 2-I-9 | 44 | 330 | 20 | 0.061 | 230.10 | 13.9 | 15.0 | | |
| 079 | 2-E-9 | 47 | 450 | 50 | 0.111 | 159.30 | 17.7 | 19.5 | -30.2% | |
| 099 | 2-I-10 | 46 | 340 | 25 | 0.074 | 197.30 | 14.5 | 15.6 | | |
| 080 | 2-E-10 | 43 | 400 | 50 | 0.125 | 136.20 | 17.0 | 18.7 | -19.4% | |
| 100 | 2-I-11 | 46 | 450 | 25 | 0.056 | | 0.0 | -1.8 | | |
| 081 | 2-E-11 | 42.5 | 420 | 50 | 0.119 | 142.40 | 17.0 | 18.6 | 1119.0% | |
| | | | | | | | | | | |
| 101 | 3-Control | 74 | 460 | 25 | 0.054 | 59.04 | 3.2 | 2.0 | | 36.5 |
| 102 | 3-I-1 | 56.5 | 490 | 50 | 0.102 | 241.15 | 24.6 | 27.8 | | |
| 103 | 3-E-1 | 60.5 | 450 | 25 | 0.056 | 73.49 | 4.1 | 3.1 | 88.9% | |
| 104 | 3-I-2 | 53 | 500 | 50 | 0.100 | 312.60 | 31.3 | 35.8 | | AVG INFL |
| 105 | 3-E-2 | 57.5 | 430 | 25 | 0.058 | 163.90 | 9.5 | 9.6 | 73.1% | 43.76054 |
| 106 | 3-I-3 | 51 | 500 | 50 | 0.100 | 345.80 | 34.6 | 39.8 | | |
| 107 | 3-E-3 | 53 | 410 | 25 | 0.061 | 248.06 | 15.1 | 16.4 | 58.8% | AVG EFFL |
| 122 | 3-I-4 | 49 | 500 | 50 | 0.100 | 283.88 | 28.4 | 32.3 | | 20.08742 |
| 123 | 3-E-4 | 49 | 430 | 25 | 0.058 | 256.17 | 14.9 | 16.1 | 50.2% | |
| 108 | 3-I-5 | 43 | 460 | 50 | 0.109 | 440.26 | 47.9 | 55.7 | | REDUCTION |
| 109 | 3-E-5 | 47 | 420 | 25 | 0.060 | 387.03 | 23.0 | 25.9 | 53.6% | 54.10% |
| 124 | 3-I-6 | 41.5 | 440 | 50 | 0.114 | 382.76 | 43.5 | 50.5 | | |
| 125 | 3-E-6 | 44 | 450 | 25 | 0.056 | 487.52 | 27.1 | 30.8 | 39.1% | |
| 110 | 3-I-7 | 42.5 | 430 | 50 | 0.116 | 474.00 | 55.1 | 64.5 | | |
| 111 | 3-E-7 | 44 | 400 | 25 | 0.063 | 540.24 | 33.8 | 38.8 | 39.8% | |
| 126 | 3-I-8 | 44 | 450 | 50 | 0.111 | 284.49 | 31.6 | 36.2 | | |
| 127 | 3-E-8 | 44 | 430 | 25 | 0.058 | 366.14 | 21.3 | 23.8 | 34.3% | |
| 138 | 3-I-9 | 51 | 490 | 50 | 0.102 | 203.40 | 20.8 | 23.1 | | |
| 139 | 3-E-9 | 55 | 370 | 25 | 0.068 | 204.00 | 13.8 | 14.8 | 36.2% | |
| | | | | | | | | | | |
| 140 | 4-Control | 73 | 470 | 25 | 0.053 | 58.51 | 3.1 | 1.9 | | 40+ |
| 141 | 4-I-1 | 65 | 500 | 25 | 0.050 | 73.74 | 3.7 | 2.6 | | |
| 142 | 4-E-1 | 70.5 | 450 | 25 | 0.056 | 32.34 | 1.8 | 0.3 | 87.0% | |
| 143 | 4-I-2 | 56 | 500 | 25 | 0.050 | 74.82 | 3.7 | 2.7 | | AVG INFL |
| 144 | 4-E-2 | 75 | 390 | 25 | 0.064 | 69.05 | 4.4 | 3.5 | -30.8% | 4.464777 |
| 145 | 4-I-3 | 63 | 460 | 25 | 0.054 | 113.87 | 6.2 | 5.6 | | |
| 146 | 4-E-3 | 74 | 400 | 25 | 0.063 | 61.32 | 3.8 | 2.8 | 50.4% | AVG EFFL |
| 147 | 4-I-4 | 64 | 500 | 25 | 0.050 | 112.20 | 5.6 | 4.9 | | 2.899505 |
| 148 | 4-E-4 | 55.5 | 400 | 25 | 0.063 | 71.97 | 4.5 | 3.6 | 27.2% | |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| 149 | 4-I-5 | 66.5 | 480 | 25 | 0.052 | 134.03 | 7.0 | 6.6 | | REDUCTION |
| 150 | 4-E-5 | 70 | 430 | 25 | 0.058 | 78.07 | 4.5 | 3.6 | 44.7% | 35.06% |
| 151 | 4-I-6 | 62 | 500 | 25 | 0.050 | 103.12 | 5.2 | 4.4 | | |
| 152 | 4-E-6 | 61 | 500 | 25 | 0.050 | 89.24 | 4.5 | 3.5 | 19.1% | |
| 153 | 4-I-7 | 62 | 370 | 25 | 0.068 | 73.17 | 4.9 | 4.1 | | |
| 154 | 4-E-7 | 62.5 | 500 | 25 | 0.050 | 74.34 | 3.7 | 2.6 | 35.8% | |
| 155 | 4-I-8 | 60 | 450 | 25 | 0.056 | 56.75 | 3.2 | 2.0 | | |
| 156 | 4-E-8 | 65.5 | 450 | 25 | 0.056 | 67.36 | 3.7 | 2.7 | -36.0% | |
| | | | | | | | | | | |
| 112 | 5-I-1 | 60.5 | 440 | 50 | 0.114 | 378.93 | 43.1 | 50.0 | | |
| 113 | 5-E-1 | 68 | 430 | 25 | 0.058 | 100.71 | 5.9 | 5.2 | 89.6% | |
| 160 | 5-I-2 | 56 | 450 | 50 | 0.111 | 234.05 | 26.0 | 29.5 | | AVG INFL |
| 161 | 5-E-2 | 67.5 | 430 | 25 | 0.058 | 157.94 | 9.2 | 9.2 | 68.7% | 47.37292 |
| 114 | 5-I-3 | 50 | 450 | 50 | 0.111 | 234.05 | 26.0 | 29.5 | | |
| 115/116 | 5-E-3 | 49.5 | 410 | 20 | 0.049 | 157.94 | 7.7 | 7.4 | 74.7% | AVG EFFL |
| 162 | 5-I-4 | 47 | 420 | 50 | 0.119 | 394.17 | 46.9 | 54.6 | | 22.13459 |
| 163 | 5-E-4 | 52 | 420 | 50 | 0.119 | 178.77 | 21.3 | 23.8 | 56.5% | |
| 164 | 5-I-5 | 44 | 420 | 50 | 0.119 | 421.47 | 50.2 | 58.5 | | REDUCTION |
| 165 | 5-E-5 | 45 | 430 | 50 | 0.116 | 274.00 | 31.9 | 36.5 | 37.6% | 53.28% |
| 166 | 5-I-6 | 42.5 | 480 | 50 | 0.104 | 511.21 | 53.3 | 62.2 | | |
| 167 | 5-E-6 | 40 | 410 | 50 | 0.122 | 357.76 | 43.6 | 50.7 | 18.6% | |
| 168 | 5-I-7 | 44.5 | 470 | 50 | 0.106 | 217.02 | 23.1 | 25.9 | | |
| 169 | 5-E-7 | 47 | 380 | 50 | 0.132 | 180.91 | 23.8 | 26.8 | -3.3% | |
| 170 | 5-I-8 | 45 | 460 | 25 | 0.054 | 229.15 | 12.5 | 13.2 | | |
| 171 | 5-E-8 | 47 | 400 | 25 | 0.063 | 189.85 | 11.9 | 12.4 | 5.4% | |
| | | | | | | | | | | |
| 117 | 6-Control | 70 | 450 | 25 | 0.056 | 21.08 | 1.2 | -0.4 | | 15 |
| 118 | 6-I-1 | 65 | 470 | 50 | 0.106 | 294.80 | 31.4 | 35.9 | | |
| 119 | 6-E-1 | 70 | 380 | 25 | 0.066 | 53.35 | 3.5 | 2.4 | 93.3% | |
| 002 | 6-I-2 | 48.5 | 450 | 50 | 0.111 | 185.43 | 20.6 | 23.0 | | AVG INFL |
| 003 | 6-E-2 | 59 | 400 | 25 | 0.063 | 66.05 | 4.1 | 3.1 | 86.3% | 28.09124 |
| 120 | 6-I-3 | 61.5 | 440 | 50 | 0.114 | 304.95 | 34.7 | 39.9 | | |
| 121 | 6-E-3 | 72 | 380 | 25 | 0.066 | 121.92 | 8.0 | 7.8 | 80.4% | AVG EFFL |
| 004 | 6-I-4 | 48.5 | 420 | 50 | 0.119 | 155.30 | 18.5 | 20.4 | | 4.258486 |
| 005 | 6-E-4 | 42 | 370 | 25 | 0.068 | 65.12 | 4.4 | 3.5 | 83.0% | |
| 006 | 6-I-5 | 49.5 | 460 | 50 | 0.109 | 207.71 | 22.6 | 25.3 | | REDUCTION |
| 007 | 6-E-5 | 68 | 430 | 25 | 0.058 | 76.84 | 4.5 | 3.6 | 86.0% | 84.84% |
| 008 | 6-I-6 | 44 | 460 | 50 | 0.109 | 198.23 | 21.5 | 24.1 | | |
| 009 | 6-E-6 | 73 | 390 | 25 | 0.064 | 90.60 | 5.8 | 5.2 | 78.6% | |
| 010 | 6-I-7 | 69 | 430 | 25 | 0.058 | 38.77 | 2.3 | 0.9 | | |
| 011 | 6-E-7 | 72.5 | 380 | 25 | 0.066 | 32.01 | 2.1 | 0.7 | 20.0% | |
| 012 | 6-I-8 | 70.5 | 400 | 25 | 0.063 | 32.07 | 2.0 | 0.6 | | |
| 013 | 6-E-8 | 74 | 410 | 25 | 0.061 | 37.60 | 2.3 | 0.9 | -58.9% | |
| | | | | | | | | | | |
| 128 | 7-Control | 73.5 | 450 | 25 | 0.056 | 48.53 | 2.7 | 1.4 | | 14.5 |
| 129 | 7-I-1 | 63 | 440 | 50 | 0.114 | 274.71 | 31.2 | 35.7 | | |
| 130 | 7-E-1 | 72.5 | 470 | 25 | 0.053 | 104.98 | 5.6 | 4.9 | 86.3% | |
| | 7-I-2 | 51 | 460 | 50 | 0.109 | 156.01 | 17.0 | 18.6 | | AVG INFL |
| | 7-E-2 | 68 | 400 | 25 | 0.063 | 86.15 | 5.4 | 4.7 | 74.9% | 27.14319 |
| 131 | 7-I-3 | 64 | 440 | 50 | 0.114 | 328.08 | 37.3 | 43.0 | | |
| 132 | 7-E-3 | 68 | 420 | 25 | 0.060 | 136.87 | 8.1 | 8.0 | 81.5% | AVG EFFL |
| | 7-I-4 | 50 | 470 | 50 | 0.106 | 197.05 | 21.0 | 23.4 | | 5.5167 |
| | 7-E-4 | 69 | 420 | 25 | 0.060 | 115.63 | 6.9 | 6.5 | 72.4% | |
| | 7-I-5 | 50 | 490 | 50 | 0.102 | 170.18 | 17.4 | 19.1 | | REDUCTION |
| | 7-E-5 | 68.5 | 480 | 25 | 0.052 | 111.68 | 5.8 | 5.2 | 72.9% | 79.68% |
| | 7-I-6 | 52 | 430 | 50 | 0.116 | 178.16 | 20.7 | 23.1 | | |
| | 7-E-6 | 67 | 470 | 25 | 0.053 | 90.17 | 4.8 | 3.9 | 82.9% | |
| | 7-I-7 | 74 | 460 | 25 | 0.054 | 32.27 | 1.8 | 0.3 | | |
| | 7-E-7 | 75 | 400 | 25 | 0.063 | 54.61 | 3.4 | 2.3 | -694.6% | |
| | 7-I-8 | 75 | 460 | 25 | 0.054 | 53.75 | 2.9 | 1.7 | | |
| | 7-E-8 | 75 | 420 | 25 | 0.060 | 30.70 | 1.8 | 0.4 | | |
| | | | | | | | | | | |
| 133 | 8-Control | 72.5 | 450 | 25 | 0.056 | 25.57 | 1.4 | -0.1 | | 15 |
| 134 | 8-I-1 | 56.5 | 460 | 100 | 0.217 | 340.33 | 74.0 | 87.2 | | |
| 135 | 8-E-1 | 69 | 450 | 25 | 0.056 | 133.03 | 7.4 | 7.1 | 91.9% | |
| | 8-I-2 | 50.5 | 450 | 50 | 0.111 | 288.44 | 32.0 | 36.7 | | AVG INFL |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 8-E-2 | 74 | 460 | 25 | 0.054 | 141.69 | 7.7 | 7.4 | 79.7% | 52.83905 |
| 136 | 8-I-3 | 57 | 450 | 100 | 0.222 | 300.33 | 66.7 | 78.4 | | |
| 137 | 8-E-3 | 65.5 | 420 | 50 | 0.119 | 179.99 | 21.4 | 23.9 | 69.5% | AVG EFFL |
| | 8-I-4 | 52.5 | 430 | 50 | 0.116 | 221.53 | 25.8 | 29.2 | | 11.33795 |
| | 8-E-4 | 67 | 440 | 25 | 0.057 | 178.56 | 10.1 | 10.4 | 64.4% | |
| | 8-I-5 | 63 | 230 | 50 | 0.217 | 194.71 | 42.3 | 49.1 | | REDUCTION |
| | 8-E-5 | 57 | 420 | 25 | 0.060 | 148.40 | 8.8 | 8.8 | 82.1% | 78.54% |
| | 8-I-6 | 55 | 460 | 50 | 0.109 | 292.84 | 31.8 | 36.5 | | |
| | 8-E-6 | 58 | 420 | 25 | 0.060 | 170.60 | 10.2 | 10.4 | 71.5% | |
| | 8-I-7 | 73.5 | 400 | 25 | 0.063 | 49.01 | 3.1 | 1.9 | | |
| | 8-E-7 | 74 | 420 | 25 | 0.060 | 54.42 | 3.2 | 2.1 | -11.4% | |
| | 8-I-8 | 74 | 430 | 25 | 0.058 | 31.00 | 1.8 | 0.3 | | |
| | 8-E-8 | 74 | 430 | 25 | 0.058 | 47.65 | 2.8 | 1.5 | -336.8% | |

AVERAGE ON-STREAM (#6/7/8)

| | |
|---|---|
| INFLUENT | 36.02 |
| EFFLUENT | 7.04 |
| REDUCTION | 80.46% |

Surface Tension on:

| Idle Smp. | 10/20/1999 | 10/21/1999 | 10/22/1999 | 10/27/1999 | 11/4/1999 |
|---|---|---|---|---|---|
| 2-EF-1* | 44 | 43 | 43 | 46.5 | 45.5 |
| 2-EF-2* | 43 | 43.5 | 44 | 45.5 | 50 |
| 2-EF-3** | 45.5 | 45.5 | 46 | 46 | 48.5 |
| 2-EF-4** | 44.5 | 46 | 47.5 | 42 | 52 |
| | | | | | |
| 3-EF-1* | | 40.5 | 48 | 41.5 | 43 |
| 3-EF-2* | | 43 | 42.5 | 41 | 47 |
| 3-EF-3** | | 43 | 52.5 | 38 | 47 |
| 3-EF-4** | | 42 | 48 | 48 | 46 |
| | | | | | |
| 4-EF-1* | | 47 | 65 | 65 | 71 |
| 4-EF-2* | | 52 | 62.5 | 66 | 68 |
| 4-EF-3** | | 51 | 61 | 65 | 62 |
| 4-EF-4** | | 56 | 66 | 69.5 | 74 |

\* samples at 20C
** samples at ambient temps.

**Appendix D:**

**Bench-scale Tests with**
**Dispersant Vs. Surface Tension Calibration**

# OHMSETT LAB TEST PLAN
## Pre-Co-Floc Removal of Dispersed Oil and Dissolved Dispersant from Ohmsett Water

1. IR Standards Preparation

   a) carefully prepare a set of $CCl_4$ standards with 200 and 500 mg/L (10.8 and 27.1 μL in 50 mL of $CCl_4$, respectively)[1] of Maya crude containing 1 part in 20 (vol:vol) Corexit 9500

   b) zero IR with pure solvent and calibrate IR with samples prepared in a)

   c) prepare an extraction efficiency curve by measuring the IR absorption of extracts from Ohmsett water samples prepared with 0, 20 and 50 mg/L of Maya crude containing 1 part in 20 (vol:vol) Corexit 9500

2. Benchmark Tests to Confirm Ottawa Results

   a) pre-coat of PB-100M — - stir 6 g of Pre-Co-Floc PB-100M into 200 mL of tank water

   - filter onto a 5.5 cm ⌀ filter paper placed in a 56 mm ⌀ceramic Buchner funnel

   b) filtration test — - prepare 50 mg/L contaminated water sample by mixing 27.1 μL[1] of Maya crude containing 1 part in 20 (vol:vol) Corexit 9500 into 500 mL of tank water; shake vigorously

   - filter contaminated water slowly through pre-coated filter, at about 200 mL/min

   c) analyse filtrate for oil - measure concentration of oil in filtered water using calibrated IR

3. Tests with Realistic Pre-Coat Thickness

   a) repeat tests described in 1. above with a pre-coat of 0.8 g of PB-100M

   b) repeat tests described in 1. above with a pre-coat of 0.8 g of PB-200M

   c) repeat a) and b) with 1.6g and 3.2 g pre-coats

4. Dissolved Dispersant Calibration Curve

   a) prepare solutions of Corexit 9500 in tank water with Corexit 9500 concentrations of: 0, 1, 2, 5,10, 20 and 50 mg/L.[1]

   b) measure water/air surface tension of solutions prepared in a) using Fisher Tensiomat

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[1] μL of contaminant required = [(desired concentration in mg/L) x (solution volume in L)/ (density of contaminant in g/mL)]: density of Maya = 0.922; density of Corexit 9500 = 0.947

c) plot surface tension of tank water vs. concentration of Corexit 9500

5. Larger Volume Tests with Pre-Co-Floc

a) prepare two 10L volumes of a 50 mg/L solution of Maya crude containing 1 part in 20 (vol:vol) Corexit 9500 in tank water

b) prepare a pre-coated filter with 1.6 g of PB-100M

c) filter one 10L sample of contaminated water prepared in a) at approx. 200 mL/min; empty filtration flask as required

d) take 500 mL samples of filtered water at approx. 5, 10, 20, 40 and at end (record time interval over which the 500 mL sample collected)

e) analyse 500 mL samples for oil content (IR) and dispersant concentration (Surface Tension)

f) repeat from b) through e) with PB-200M

Corexit 9500:Maya Crude
1: 20
50 mg/l in 500 mLs Ohmsett tank $H_2O$
Filtered through PreCoFloc @ approx. 200 mL/min

| Mass (g) | Rep # | PB-100M Total TPH | Avg. | Corrected* | Avg. Corrected* | PB-200M Total TPH | Avg. | Corrected* | Avg. Corrected* |
|---|---|---|---|---|---|---|---|---|---|
| 6 | 1 | 3.22 | | 2.05 | | -0.05 | | -1.88 | |
| | 2 | 2.52 | | 1.21 | | -0.17 | | -2.03 | |
| | 3 | 4.34 | 3.36 | 3.40 | 2.22 | -0.33 | -0.18 | -2.22 | -2.04 |
| 3.2 | 1 | -0.53 | | -2.46 | | 2.26 | | 0.90 | |
| | 2 | -0.58 | | -2.52 | | -0.55 | | -2.48 | |
| | 3 | -0.49 | -0.53 | -2.41 | -2.46 | -0.07 | 0.55 | -1.91 | -1.16 |
| 1.6 | 1 | -0.23 | | -2.10 | | 9.72 | | 9.87 | |
| | 2 | 2.37 | | 1.03 | | 0.69 | | -0.99 | |
| | 3 | 1.82 | 1.32 | 0.37 | -0.23 | 2.21 | 4.21 | 0.84 | 3.24 |
| 0.8 | 1 | 5.23 | | 4.47 | | 5.44 | | 4.72 | |
| | 2 | 6.63 | | 6.15 | | 2.23 | | 0.86 | |
| | 3 | 3.46 | 5.11 | 2.34 | 4.32 | 3.07 | 3.58 | 1.87 | 2.48 |

* Corrected using extraction efficiency correlation

Surface Tension vs. Dispersant Concentration

T = 24C

| mg/L | Rep 1 | Rep 2 | Rep 3 | Rep 4 | Rep 5 | Avg. | Best Fit Curve |
|---|---|---|---|---|---|---|---|
| DI=Control | 70.4 | 71 | 69.8 | | | 70.4 | |
| 0 | 73 | 73.3 | 73.1 | | | 73.1 | 72.02223 |
| 1 | 64.9 | 66.8 | 60.5 | 64.7 | 71 | 65.6 | 69.6165 |
| 2 | 71.4 | 69.3 | 70.5 | 65 | 71.1 | 69.5 | 67.391 |
| 5 | 65 | 66.7 | 64.8 | 58.3 | 65.5 | 64.1 | 61.666 |
| 10 | 56.3 | 53.2 | 51.2 | 51.2 | 52.8 | 52.9 | 54.651 |
| 20 | 46.5 | 46.5 | 46.6 | 45.4 | 47.8 | 46.6 | 46.6799 |
| 50 | 40.6 | 40.8 | 40.9 | 40.4 | 41.1 | 40.8 | 40.5736 |



## ST vs. Dispersant Conc.

$ST = 39.9206 + 32.1017e^{(-0.0779 \times Conc.)}$

OR

$Conc. = -12.837\ln([ST-39.9206]/32.1017)$

$R^2 = 0.9658$

Legend: Rep 1, Rep 2, Rep 3, Rep 4, Rep 5, Rep 6, Best Fit Curve

Y-axis: Surface Tension (dynes/cm)

X-axis: Dispersant Conc. (mg/L)
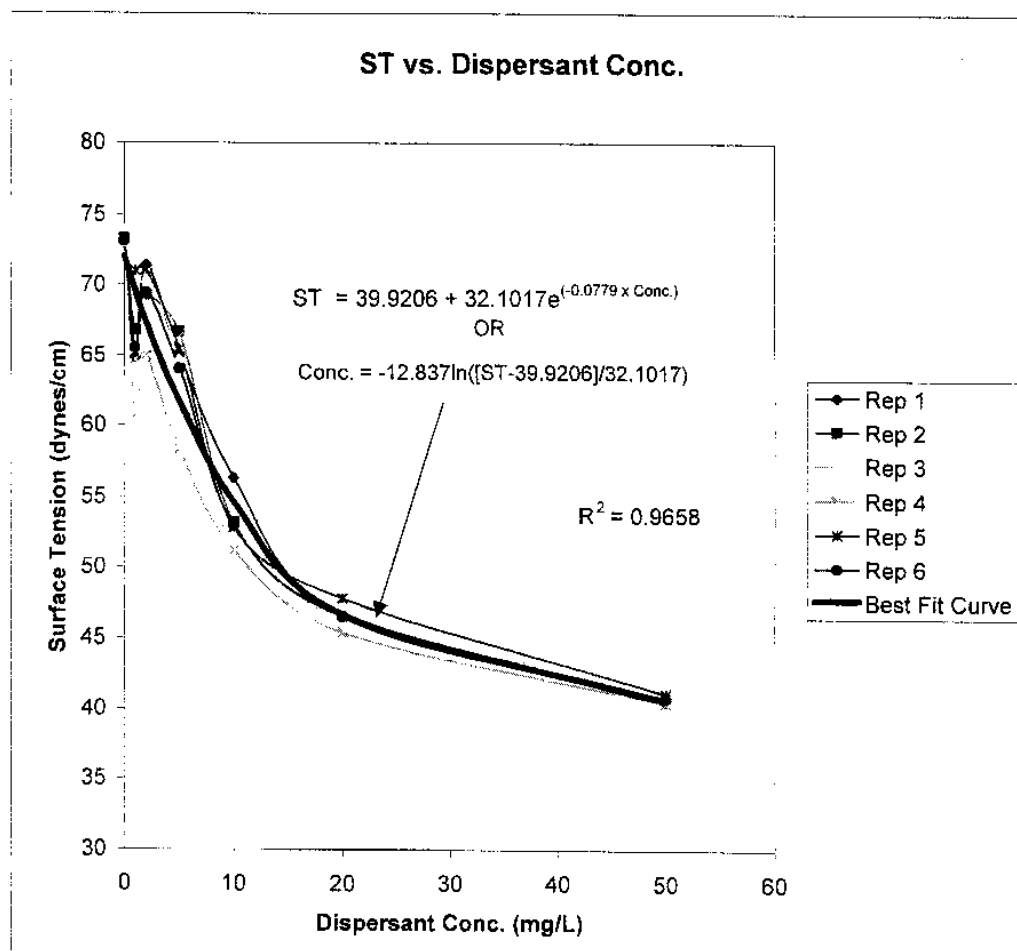
10L Tank H2O w/ .542 mls Maya crude (w/ 1:20 Corexit) = 50 mg/L total. or 2.5 mg/L 9500 and 47.5 mg/L Maya
Filtered thru 1.6 g PB-100M, PB-200M
Took samples every 5,10,20,30,40 minutes & at end

## Round #1

### PB-100M

| Std Conc. | TPH Rdg. | Smp. Vol | Extr. Vol | Extr/Smp | TTL TPH | Corrected+ | Calc. 9500 conc. (mg/L) | Avg ST | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Std 0 | -7.37 | | | | | | | | | | | |
| Std 200 | 203.59 | | | | | | | | | | | |
| Std 500 | 499.12 | | | | | | | | | | | |
| Time (min) | | | | | | | | | | | | |
| 5 | -4.02 | 100 | 50 | 0.5 | -2.01 | -4.24 | 1.71 | 68.0 | 63.3 | 70.5 | 67.5 | 70.8 |
| 10 | -5.18 | 100 | 50 | 0.5 | -2.59 | -4.94 | 0.57 | 70.6 | 70.4 | 72.9 | 71.4 | 67.8 |
| 20 | -5.17 | 100 | 50 | 0.5 | -2.59 | -4.93 | 4.53 | 62.5 | 57.9 | 64.9 | 67.4 | 59.7 |
| 30 | -3.94 | 100 | 50 | 0.5 | -1.97 | -4.19 | 1.27 | 69.0 | 70 | 68.6 | 68.4 | |
| 40 | -2.47 | 100 | 50 | 0.5 | -1.24 | -3.31 | 2.72 | 65.9 | 62.7 | 65.5 | 69.5 | |
| End = 75 | -4.28 | 100 | 50 | 0.5 | -2.14 | -4.40 | 4.18 | 63.1 | 58.4 | 65 | 67.8 | 61.2 |

### PB-200M

| Time (min) | TPH Rdg. | Smp. Vol | Extr. Vol | Extr/Smp | TTL TPH | Corrected+ | Calc. 9500 conc. (mg/L) | Avg ST | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 5 | 85.84 | 100 | 50 | 0.5 | 42.92 | 49.80 | 2.52 | 66.3 | 62.8 | 65.7 | 67.7 | 69 |
| 10 | 26.34 | 100 | 50 | 0.5 | 13.17 | 14.02 | 5.05 | 61.6 | 58 | 67.6 | 56 | 64.7 |
| 20 | 19.7 | 100 | 50 | 0.5 | 9.85 | 10.02 | 8.46 | 56.5 | 51.8 | 56.8 | 61 | |
| 30 | -1.67 | 100 | 50 | 0.5 | -0.84 | -2.83 | 5.57 | 60.7 | 57.9 | 62.9 | 60.9 | 61.2 |
| 40 | -1.1 | 100 | 50 | 0.5 | -0.55 | -2.48 | 4.91 | 61.8 | 70 | 60.6 | 59.5 | 57.2 |
| End = 45 | 0.53 | 100 | 50 | 0.5 | 0.27 | -1.50 | 19.27 | 47.1 | 47.1 | 47 | 50.2 | 44 |

Repeated this series once we got comfortable with method and procedure :

## Round #2

### PB-100M

| Std Conc. | TPH Rdg. | Smp. Vol | Extr. Vol | Extr/Smp | TTL TPH | Corrected+ | Calc. 9500 conc. (mg/L) | Avg ST | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Std 0 | -16.7 | | | | | | | | | | | |
| Std 200 | 201.1 | | | | | | | | | | | |
| Std 500 | 497.3 | | | | | | | | | | | |
| Prefilter | 50.29 | 100 | 50 | 0.5 | 25.15 | 28.42 | | | | | | |
| Time (min) | | | | | | | | | | | | |
| 5 | -9.36 | 100 | 50 | 0.5 | -4.68 | -7.45 | 2.54 | 66.3 | 62.5 | 66.2 | 70.8 | 65.5 |
| 10 | -7.04 | 100 | 50 | 0.5 | -3.52 | -6.06 | 1.79 | 67.9 | 69.4 | 67.2 | 65.6 | 69.2 |
| Prefilter | 23.87 | 100 | 50 | 0.5 | 11.94 | 12.53 | 4.03 | 63.4 | | | | |
| 20 | -7.02 | 100 | 50 | 0.5 | -3.51 | -6.04 | 4.03 | 63.4 | 60 | 68.2 | 63.3 | 62 |
| 30 | 4.89 | 100 | 50 | 0.5 | 2.45 | 1.12 | 3.13 | 65.1 | 70.8 | 65.2 | 61.9 | 62.4 |
| End = 40 | 0.103 | 100 | 50 | 0.5 | 0.05 | -1.76 | 7.64 | 57.6 | 54.8 | 57.7 | 60.2 | 57.8 |

### PB-200M

| Std Conc. | TPH Rdg. | Smp. Vol | Extr. Vol | Extr/Smp | TTL TPH | Corrected+ | Calc. 9500 conc. (mg/L) | Avg ST | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Std 0 | -19.55 | | | | | | | | | | | |
| Std 200 | 196.9 | | | | | | | | | | | |
| Std 500 | 494.7 | | | | | | | | | | | |
| Prefilter* | 4.99 | 100 | 50 | 0.5 | 2.495 | 1.18 | | | | | | |
| Time (min) | | | | | | | | | | | | |
| 5 | -11.31 | 100 | 50 | 0.5 | -5.655 | -8.62 | 0.99 | 69.6 | 67.1 | 70.8 | 71 | |
| 10 | -8.91 | 100 | 50 | 0.5 | -4.455 | -7.18 | 2.72 | 65.9 | 62 | 66.3 | 68 | 67.3 |
| 20 | -5.51 | 100 | 50 | 0.5 | -2.755 | -5.14 | | | | | | |
| 30 | -7.2 | 100 | 50 | 0.5 | -3.6 | -6.15 | | | | | | |
| End = 40 | -10.3 | 100 | 50 | 0.5 | -5.15 | -8.02 | 6.13 | 59.8 | 57 | 60.6 | 61.7 | |

* Entry of 4.99 in lab book is likely an error; probably should be 49.99
+ Corrected using extraction efficiency correlation

**Appendix E:**

**Results of Tests to Measure**

**Degradation of Dispersant**

NATURAL DEGRADATION OF DISPERSANT

|  | SURFACE TENSION MEASURED ON DATE: | | | | |
|---|---|---|---|---|---|
|  | 10/20 | 10/21 | 10/22 | 10/27 | 11/04 |
| 2-EF-1* | 44 | 43 | 43 | 46.5 | 45.5 |
| 2-EF-2* | 43 | 43.5 | 44 | 45.5 | 50 |
| AVG | 43.5 | 43.25 | 43.5 | 46 | 47.75 |
| "CALCULATED" DISPERSANT CONC | 28.2 | 29.1 | 28.2 | 21.4 | 18.1 |
|  |  |  |  |  |  |
| 2-EF-3** | 45.5 | 45.5 | 46 | 46 | 48.5 |
| 2-EF-4** | 44.5 | 46 | 47.5 | 42 | 52 |
| AVG | 45 | 45.75 | 46.75 | 44 | 50.25 |
| "CALCULATED" DISPERSANT CONC | 23.7 | 21.9 | 19.9 | 26.5 | 14.6 |
|  |  |  |  |  |  |
| 3-EF-1* |  | 40.5 | 48 | 41.5 | 43 |
| 3-EF-2* |  | 43 | 42.5 | 41 | 47 |
| AVG |  | 41.75 | 45.25 | 41.25 | 45 |
| "CALCULATED" DISPERSANT CONC |  | 36.8 | 23.1 | 40.9 | 23.7 |
|  |  |  |  |  |  |
| 3-EF-3** |  | 43 | 52.5 | 38 | 47 |
| 3-EF-4** |  | 42 | 48 | 48 | 46 |
| AVG |  | 42.5 | 50.25 | 43 | 46.5 |
| "CALCULATED" DISPERSANT CONC |  | 32.4 | 14.6 | 30.1 | 20.3 |
|  |  |  |  |  |  |
| 4-EF-1* |  | 47 | 65 | 65 | 71 |
| 4-EF-2* |  | 52 | 62.5 | 66 | 68 |
| AVG |  | 49.5 | 63.75 | 65.5 | 69.5 |
| "CALCULATED" DISPERSANT CONC |  | 15.5 | 3.8 | 2.9 | 1.1 |
|  |  |  |  |  |  |
| 4-EF-3** |  | 51 | 61 | 65 | 62 |
| 4-EF-4** |  | 56 | 66 | 69.5 | 74 |
| AVG |  | 53.5 | 63.5 | 67.25 | 68 |
| "CALCULATED" DISPERSANT CONC |  | 11.0 | 4.0 | 2.1 | 1.7 |

* samples at 20C
** samples at outdoor ambient temperature
"CALCULATED" DISPERSANT CONC: calculated using dispersant concentration vs.
  surface tension calibration in Appendix D

# Extraction Efficiency Correlation



$y = 1.2027x - 1.822$

$R^2 = 0.9978$

Measured TPH (mg/L)

Actual TPH (mg/L)